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METHOD 8081

ORGANOCHLORINE PESTICIDES AND PCBs AS AROCLORS BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE

1.0 SCOPE AND APPLICATION

1.1 Method 8081 is used to determine the concentrations of various organochlorine pesticides and polychlorinated biphenyls (PCBs) as Aroclors, in extracts from solid and liquid matrices. Open-tubular, capillary columns were employed with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). When compared to the packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The list below is annotated to show whether a single- or dual-column analysis system was used to identify each target analyte.

Compound Name	CAS Registry No.
Aldrin[a,b]	309-00-2
Aroclor-1016[a,b]	12674-11-2
Aroclor-1221[a,b]	1104-28-2
Aroclor-1232[a,b]	11141-16-5
Aroclor-1242[a,b]	53469-21-9
Aroclor-1248[a,b]	12672-29-6
Aroclor-1254[a,b]	11097-69-1
Aroclor-1260[a,b]	11096-82-5
α-BHC[a,b]	319-84-6
β-BHC[a,b]	319-85-7
γ-BHC (Lindane)[a,b]	58-89-9
δ-BHC[a,b]	319-86-8
Chlorobenzilate[b]	510-15-6
α-Chlordane[b]	5103-71-9
γ-Chlordane[a,b]	5103-74-2
DBCP[b]	96-12-8
4,4'-DDD[a,b]	72-54-8
4,4'-DDE[a,b]	72-55-9
4,4'-DDT[a,b]	50-29-3
Diallate[b]	2303-16-4
Dieldrin[a,b]	60-57-1
Endosulfan I[a,b]	959-98-8
Endosulfan II[a,b]	33213-65-9
Endosulfan sulfate[a,b]	1031-07-8
Endrin[a,b]	72-20-8
Endrin aldehyde[a,b]	7421-93-4
Endrin ketone[b]	53494-70-5

Compound Name	CAS Registry No.
Heptachlor[a,b]	76-44-8
Heptachlor epoxide[a,b]	1024-57-3
Hexachlorobenzene[b]	118-74-1
Hexachlorocyclopentadiene[b]	77-47-4
Isodrin[b]	465-73-6
Kepone[b]	143-50-0
Methoxychlor[a,b]	72-43-5
Toxaphene[a,b]	8001-35-2

[a] Single-column analysis
[b] Dual-column analysis

1.2 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.).

1.3 Although performance data are presented for many of the listed chemicals, it is unlikely that all of them could be determined in a single analysis. This limitation results because the chemical and chromatographic behavior of many of these chemicals can result in co-elution. Several cleanup/fractionation schemes are provided in this method and in Method 3600. Any chemical is a potential method interference when it is not a target analyte.

1.4 Several multi-component mixtures (i.e., Aroclors and Toxaphene) are listed as target compounds. When samples contain more than one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation or degradation by treatment technologies. These result in "weathered" Aroclors (or any other multi-component mixtures) that may have significant differences in peak patterns than those of standards. In these cases, individual congener analyses may be preferred over total mixture analyses.

1.5 Compound identification based on single column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS Method 8270 is also recommended as a confirmation technique if sensitivity permits (Sec. 8).

1.6 This method describes a dual column option. The option allows a hardware configuration of two analytical columns joined to a single injection port. The option allows one injection to be used for dual column analysis.

Analysts are cautioned that the dual column option may not be appropriate when the instrument is subject to mechanical stress, many samples are to be run in a short period, or when contaminated samples are analyzed.

1.7 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.8 Extracts suitable for analysis by this method may also be analyzed for organophosphorus pesticides (Method 8141). Some extracts may also be suitable for triazine herbicide analysis, if low recoveries (normally samples taken for triazine analysis must be preserved) are not a problem.

1.9 The following compounds may also be determined using this method:

Compound Name	CAS Registry No.
Alachlor[a,b]	15972-60-8
Captafol[b]	2425-06-1
Captan[b]	133-06-2
Chloroneb[b]	2675-77-6
Chloropropylate[b]	99516-95-7
Chlorothalonil[b]	1897-45-6
DCPA[b]	1861-32-1
Dichlone[b]	117-80-6
Dicofol[b]	115-32-2
Etridiazole[b]	2593-15-9
Halowax-1000[b]	58718-66-4
Halowax-1001[b]	58718-67-5
Halowax-1013[b]	12616-35-2
Halowax-1014[b]	12616-36-3
Halowax-1051[b]	2234-13-1
Halowax-1099[b]	39450-05-0
Mirex[b]	2385-85-5
Nitrofen[b]	1836-75-5
PCNB[b]	82-68-8
Perthane[b]	72-56-0
Propachlor[b]	1918-16-17
Strobane[b]	8001-50-1
trans-Nonachlor[b]	39765-80-5
trans-Permethrin[b]	51877-74-8
Trifluralin[b]	1582-09-8

[a] Single-column analysis
[b] Dual-column analysis

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate sample extraction technique. Liquid samples are extracted at neutral pH with methylene chloride using either a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520). Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using either Soxhlet extraction (Method 3540), Automated Soxhlet (Method 3541), or Ultrasonic Extraction (Method 3550). A variety of cleanup steps may be applied to the extract, depending on (1) the nature of the coextracted matrix interferences and (2) the target analytes. After cleanup, the extract is analyzed by injecting a 1-μL sample into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD) or an electrolytic conductivity detector (GC/ELCD).

3.0 INTERFERENCES

3.1 Refer to Methods 3500 (Sec. 3, in particular), 3600, and 8000.

3.2 Sources of interference in this method can be grouped into three broad categories: contaminated solvents, reagents or sample processing hardware; contaminated GC carrier gas, parts, column surfaces or detector surfaces; and the presence of coeluting compounds in the sample matrix to which the ECD will respond. Interferences coextracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

3.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. These materials may be removed prior to analysis using Gel Permeation Cleanup - pesticide option (Method 3640) or as Fraction III of the silica gel cleanup procedure (Method 3630). Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

3.4 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry in an oven at 130°C for several hours or rinse with methanol and drain. Store dry glassware in a clean environment.

3.5 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur

contamination should be expected with sediment samples. Method 3660 is suggested for removal of sulfur. Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup.

3.6 Waxes, lipids, and other high molecular weight co-extractables can be removed by Gel-Permeation Cleanup (Method 3640).

3.7 It may be difficult to quantitate Aroclor patterns and single component pesticides together. Some pesticides can be removed by sulfuric acid/permanganate cleanup (Method 3665) and silica fractionation (Method 3630). Guidance on the identification of PCBs is given in Sec. 7.

3.8 The following target analytes coelute using single column analysis:

DB 608	Trifluralin/Diallate isomers
	PCNP/Dichlone/Isodrin
	DDD/Endosulfan II
DB 1701	Captan/Chlorobenzilate
	Captafol/Mirex
	DDD/Endosulfan II
	Methoxychlor/Endosulfan sulfate

3.8.1 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphorus pesticides are eliminated by the Gel Permeation Chromatography cleanup - pesticide option (Method 3640). Co-eluting chlorophenols are eliminated by Silica gel (Method 3630), Florisil (Method 3620), or Alumina (Method 3610) cleanup.

3.9 The following compounds coelute using the dual column analysis. Two temperature programs are provided for the same pair of columns as option 1 and option 2 for dual column analysis. In general, the DB-5 column resolves fewer compounds that the DB-1701:

3.9.1 DB-5/DB-1701, thin film, slow ramp: See Sec. 7 and Table 6.

DB-5	*trans*-Permethrin/Heptachlor epoxide
	Endosulfan I/*α*-Chlordane
	Perthane/Endrin
	Endosulfan II/Chloropropylate/Chlorobenzilate
	4,4'-DDT/Endosulfan sulfate
	Methoxychlor/Dicofol

Perthane/Endrin and Chlorobenzilate/Endosulfan II/Chloropropylate will also co-elute on DB-5 after moderate deterioration in column performance.

DB-1701 Chlorothalonil/ß-BHC
δ-BHC/DCPA/*trans*-Permethrin
α-Chlordane/*trans*-Nonachlor
Captan/Dieldrin
Chlorobenzilate/Chloropropylate

Chlorothalonil/ß-BHC and *α*-Chlordane/*trans*-Nonachlor will co-elute on the DB-1701 column after moderate deterioration in column performance.

Nitrofen, Dichlone, Carbophenothion, Dichloran and Kepone were removed from the composite mixture because of extensive peak tailing on both columns. Simazine and Atrazine give poor responses on the ECD detector. Triazine compounds should be analyzed using Method 8141 (NPD option).

3.9.2 DB-5/DB-1701, thick film, fast ramp: See Sec. 7 and Table 7.

DB-5 Diallate/*α*-BHC
Perthane/Endosulfan II
Chlorobenzilate/Chloropropylate
Endrin/Nitrofen
4,4'-DDT/Endosulfan sulfate
Methoxychlor/Dicolfol

DB-1701 *α*-Chlordane/*trans*-Nonachlor (partially resolved)
4,4'-DDD/Endosulfan II (partially resolved)

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: an analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and recorder/integrator or data system.

The columns listed in this section were used to develop the method performance data. Their specification is not intended to prevent laboratories from using columns that are developed after promulgation of the method. Laboratories may use other capillary columns if they document method performance data (e.g. chromatographic resolution, analyte breakdown, and MDLs) equal to or better than those provided with the method.

4.1.1 Single-column Analysis:

4.1.1.1 Narrow-bore columns:

4.1.1.1.1 Column 1 - 30 m x 0.25 or 0.32 mm internal diameter (ID) fused silica capillary column chemically bonded with SE-54 (DB 5 or equivalent), 1 μm film thickness.

4.1.1.1.2 Column 2 - 30 m x 0.25 mm ID fused silica capillary column chemically bonded with 35 percent phenyl

methylpolysiloxane (DB 608, SPB 608, or equivalent), 25 μm coating thickness, 1 μm film thickness.

4.1.1.1.3 Narrow bore columns should be installed in split/splitless (Grob-type) injectors.

4.1.1.2 Wide-bore columns

4.1.1.2.1 Column 1 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB 608, SPB 608, RTx-35, or equivalent), 0.5 μm or 0.83 μm film thickness.

4.1.1.2.2 Column 2 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB 1701, or equivalent), 1.0 μm film thickness.

4.1.1.2.3 Column 3 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with SE-54 (DB 5, SPB 5, RTx5, or equivalent), 1.5 μm film thickness.

4.1.1.2.4 Wide-bore columns should be installed in 1/4 inch injectors, with deactivated liners designed specifically for use with these columns.

4.1.2 Dual Column Analysis:

4.1.2.1 Column pair 1:

4.1.2.1.1 J&W Scientific press-fit Y-shaped glass 3-way union splitter (J&W Scientific, Catalog no. 705-0733) or Restek Y-shaped fused-silica connector (Restek, Catalog no. 20405), or equivalent.

4.1.2.1.2 30 m x 0.53 m ID DB-5 (J&W Scientific), 1.5 μm film thickness, or equivalent.

4.1.2.1.3 30 m x 0.53 mm ID DB-1701 (J&W Scientific), 1.0 μm film thickness, or equivalent.

4.1.2.2 Column pair 2:

4.1.2.2.1 Splitter 2 - Supelco 8 in. glass injection tee, deactivated (Supelco, Catalog no. 2-3665M), or equivalent.

4.1.2.2.2 30 m x 0.53 m ID DB-5 (J&W Scientific), 0.83 μm film thickness, or equivalent.

4.1.2.2.3 30 m x 0.53 mm ID DB-1701 (J&W Scientific), 1.0 μm film thickness, or equivalent.

4.1.3 Column rinsing kit: Bonded-phase column rinse kit (J&W Scientific, Catalog no. 430-3000 or equivalent).

4.2 Glassware (see Methods 3510, 3520, 3540, 3541, 3550, 3630, 3640, 3660, and 3665 for specifications).

4.3 Kuderna-Danish (K-D) apparatus. See extraction methods for specifics.

5.0 REAGENTS

5.1 Reagent or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4°C in Teflon-sealed containers in the dark. When a lot of standards is prepared, it is recommended that aliquots of that lot be stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC (Sec. 8) indicates a problem. All other standard solutions must be replaced after six months or sooner if routine QC (Sec. 8) indicates a problem.

5.2 Solvents and reagents: As appropriate for Method 3510, 3520, 3540, 3541, 3550, 3630, 3640, 3660, or 3665: n-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane). All solvents should be pesticide quality or equivalent, and each lot of solvent should be determined to be phthalate free. Solvents must be exchanged to hexane or isooctane prior to analysis.

5.2.1 Organic-free reagent water: All references to water in this method refer to organic-free reagent water as defined in Chapter One.

5.3 Stock standard solutions (1000 mg/L): Can be prepared from pure standard materials or can be purchased as certified solutions.

5.3.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure compound. Dissolve the compound in isooctane or hexane and dilute to volume in a 10-mL volumetric flask. If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution. Commercially prepared stock standard solutions can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.2 ß-BHC, Dieldrin, and some other standards may not be adequately soluble in isooctane. A small amount of acetone or toluene should be used to dissolve these compounds during the preparation of the stock standard solutions.

5.4 Composite stock standard: Can be prepared from individual stock solutions. For composite stock standards containing less than 25 components, take exactly 1 mL of each individual stock solution at 1000 mg/L, add solvent, and mix the solutions in a 25-mL volumetric flask. For example, for a composite containing 20 individual standards, the resulting concentration of each component in the mixture, after the volume is adjusted to 25 mL, will be 1 mg/25 mL. This composite solution can be further diluted to obtain the desired concentrations. For composite stock standards containing more than 25 components, use volumetric flasks of the appropriate volume (e.g., 50 mL, 100 mL).

5.5 Calibration standards should be prepared at a minimum of five concentrations by dilution of the composite stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector.

5.5.1 Although all single component analytes can be resolved on a new 35 percent phenyl methyl silicone column (e.g., DB-608), two calibration mixtures should be prepared for the single component analytes of this method.

5.5.2 This procedure is established to (1) minimize potential resolution and quantitation problems on confirmation columns or on older 35 percent phenyl methyl silicone (e.g. DB-608) columns and (2) allow determination of Endrin and DDT breakdown for method QC (Sec. 8).

5.5.3 Separate calibration standards are required for each multi-component target analyte, with the exception of Aroclors 1016 and 1260, which can be run as a mixture.

5.6 Internal standard (optional):

5.6.1 Pentachloronitrobenzene is suggested as an internal standard for the single column analysis, when it is not considered to be a target analyte. 1-Bromo-2-nitrobenzene is a suggested option. Prepare the standard to complement the concentrations found in Sec. 5.5.

5.6.2 Make a solution of 1000 mg/L of 1-bromo-2-nitrobenzene for dual-column analysis. Dilute it to 500 ng/μL for spiking, then use a spiking volume of 10 μL/mL of extract.

5.7 Surrogate standards: The performance of the method should be monitored using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards.

5.7.1 For the single column analysis, use decachlorobiphenyl as the primary surrogate. However, if recovery is low, or late-eluting compounds interfere with decachlorobiphenyl, then tetrachloro-m-xylene should be evaluated as a surrogate. Proceed with corrective action when both surrogates are out of limits for a sample (Sec. 8.2). Method 3500, Sec. 5, indicates the proper procedure for preparing these surrogates.

5.7.2 For the dual column analysis make a solution of 1000 mg/L of 4-chloro-3-nitrobenzotrifluoride and dilute to 500 ng/μL. Use a spiking volume of 100 μL for a 1 L aqueous sample. Store the spiking solutions at 4°C in Teflon-sealed containers in the dark.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 See Chapter 4, Organic Analytes, Sec. 4.

6.2 Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

7.1.1 Refer to Chapter Two and Method 3500 for guidance in choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520). Extract solid samples with hexane-acetone (1:1) using one of the Soxhlet extraction (Method 3540 or 3541) or ultrasonic extraction (Method 3550) procedures.

NOTE: Hexane/acetone (1:1) may be more effective as an extraction solvent for organochlorine pesticides and PCBs in some environmental and waste matrices than is methylene chloride/acetone (1:1). Use of hexane/acetone generally reduces the amount of co-extracted interferences and improves signal/noise.

7.1.2 Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. Each sample type must be spiked with the compounds of interest to determine the percent recovery and the limit of detection for that sample (Sec. 5). See Method 8000 for guidance on demonstration of initial method proficiency as well as guidance on matrix spikes for routine sample analysis.

7.2 Cleanup/Fractionation:

7.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. General guidance for sample extract cleanup is provided in this section and in Method 3600.

7.2.1.1 If a sample is of biological origin, or contains high molecular weight materials, the use of GPC cleanup/pesticide option (Method 3640) is recommended. Frequently, one of the

adsorption chromatographic cleanups may also be required following the GPC cleanup.

7.2.1.2 If only PCBs are to be measured in a sample, the sulfuric acid/permanganate cleanup (Method 3665) is recommended. Additional cleanup/fractionation by Alumina Cleanup (Method 3610), Silica-Gel Cleanup (Method 3630), or Florisil Cleanup (Method 3620), may be necessary.

7.2.1.3 If both PCBs and pesticides are to be measured in the sample, isolation of the PCB fraction by Silica Cleanup (Method 3630) is recommended.

7.2.1.4 If only pesticides are to be measured, cleanup by Method 3620 or Method 3630 is recommended.

7.2.1.5 Elemental sulfur, which may appear in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur should be removed by the technique described in Method 3660, Sulfur Cleanup.

7.3 GC Conditions: This method allows the analyst to choose between a single column or a dual column configuration in the injector port. Either wide- or narrow-bore columns may be used. Identifications based on retention times from a single column must be confirmed on a second column or with an alternative qualitative technique.

7.3.1 Single Column Analysis:

7.3.1.1 This capillary GC/ECD method allows the analyst the option of using 0.25-0.32 mm ID capillary columns (narrow-bore) or 0.53 mm ID capillary columns (wide-bore). Performance data are provided for both options. Figures 1-6 provide example chromatograms.

7.3.1.2 The use of narrow-bore columns is recommended when the analyst requires greater chromatographic resolution. Use of narrow-bore columns is suitable for relatively clean samples or for extracts that have been prepared with one or more of the clean-up options referenced in the method. Wide-bore columns (0.53 mm) are suitable for more complex environmental and waste matrices.

7.3.1.3 For the single column method of analysis, using wide-bore capillary columns, Table 1 lists average retention times and method detection limits (MDLs) for the target analytes in water and soil matrices. For the single column method of analysis, using narrow-bore capillary columns, Table 2 lists average retention times and method detection limits (MDLs) for the target analytes in water and soil matrices. The MDLs for the components of a specific sample may differ from those listed in Tables 1 and 2 because they are dependent upon the nature of interferences in the sample matrix. Table 3 lists the Estimated Quantitation Limits (EQLs) for other matrices. Table 4 lists the GC operating conditions for the single column method of analysis.

7.3.2 Dual Column Analysis:

7.3.2.1 The dual-column/dual-detector approach involves the use of two 30 m x 0.53 mm ID fused-silica open-tubular columns of different polarities, thus different selectivities towards the target compounds. The columns are connected to an injection tee and ECD detectors. Retention times for the organochlorine analytes on dual columns are in Table 5. The GC operating conditions for the compounds in Table 5 are in Table 6. Multicomponent mixtures of Toxaphene and Strobane were analyzed separately (Figures 7 and 8) using the GC operating conditions found in Table 7. Seven Aroclor mixtures and six Halowax mixtures were analyzed under the conditions outlined in Table 7 (Figures 9 through 21). Figure 22 is a sample chromatogram for a mixture of organochlorine pesticides. The retention times of the individual components detected in these mixtures are given in Tables 8 and 9.

7.3.2.1.1 Operating conditions for a more heavily loaded DB-5/DB-1701 pair are given in Table 7. This column pair was used for the detection of multicomponent organochlorine compounds.

7.3.2.1.2 Operating conditions for a DB-5/DB-1701 column pair with thinner films, a different type of splitter, and a slower temperature programming rate are provided in Table 6. These conditions gave better peak shapes for compounds such as Nitrofen and Dicofol. Table 5 lists the retention times for the compounds detected on this column pair.

7.4 Calibration:

7.4.1 Prepare calibration standards using the procedures in Sec. 5. Refer to Method 8000 (Sec. 7) for proper calibration techniques for both initial calibration and calibration verification. The procedure for either internal or external calibration may be used, however, in most cases external standard calibration is used with Method 8081. This is because of the sensitivity of the electron capture detector and the probability of the internal standard being affected by interferences. Because several of the pesticides may co-elute on any single column, analysts should use two calibration mixtures (see Sec. 3.8). The specific mixture should be selected to minimize the problem of peak overlap.

NOTE: Because of the sensitivity of the electron capture detector, the injection port and column should always be cleaned prior to performing the initial calibration.

7.4.1.1 Method 8081 has many multi-component target analytes. For this reason, the target analytes chosen for calibration should be limited to those specified in the project plan. For instance, some sites may require analysis for the organochlorine pesticides only or the PCBs only. Toxaphene and/or technical Chlordane may not be specified at certain sites. In addition, where PCBs are specified in the project plan, a mixture of

Aroclors 1016 and 1260 will suffice for the initial calibration of all Aroclors, since they include all congeners present in the different regulated Aroclors. A mid-point calibration standard of all Aroclors (for Aroclor pattern recognition) must be included with the initial calibration so that the analyst is familiar with each Aroclor pattern and retention times on each column.

7.4.1.2 For calibration verification (each 12 hr shift) all target analytes required in the project plan must be injected with the following exception for the Aroclors. For sites that require PCB analysis, include only the Aroclors that are expected to be found at the site. If PCBs are required, but it is unknown which Aroclors may be present, the mid-concentration Aroclors 1016/1260 mixture only, may be injected. However, if specific Aroclors are found at the site during the initial screening, it is required that the samples containing Aroclors be reinjected with the proper mid-concentration Aroclor standards.

7.4.2 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more. Therefore, the GC column should be primed or deactivated by injecting a PCB or pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

CAUTION: Several analytes, including Aldrin, may be observed in the injection just following this system priming. Always run an acceptable blank prior to running any standards or samples.

7.4.3 Retention time windows:

7.4.3.1 Before establishing the retention time windows, make sure the gas chromatographic system is within optimum operating conditions. The width of the retention time window should be based upon actual retention times of standards measured over the course of 72 hours. See Method 8000 for details.

7.4.3.2 Retention time windows shall be defined as plus or minus three times the standard deviation of the absolute retention times for each standard. However, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. For multicomponent standards (i.e., PCBs), the analyst should use the retention time window but should primarily rely on pattern recognition. Sec. 7.5.4 provides guidance on the establishment of absolute retention time windows.

7.4.3.3 Certain analytes, particularly Kepone, are subject to changes in retention times. Dry Kepone standards prepared in hexane or isooctane can produce gaussian peaks. However, Kepone extracted from samples or standards exposed to water or methanol may produce peaks with broad tails that elute later than the standard (0-1 minute). This shift is presumably the result of the formation

of a hemi-acetal from the ketone functionality. Method 8270 is recommended for Kepone.

7.5 Gas chromatographic analysis:

7.5.1 Set up the GC system using the conditions described in Tables 4, 6, or 7. An initial oven temperature at or below 140-150°C is required to resolve the four BHC isomers. A final temperature of 240-270°C is required to elute decachlorobiphenyl. Use of injector pressure programming will improve the chromatography of late eluting peaks.

7.5.2 Verify calibration each 12 hour shift by injecting calibration verification standards prior to conducting any analyses. See Sec. 7.4.1.2 for special guidance on calibration verification of PCBs. A calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded) and at the end of the analysis sequence. The calibration factor for each analyte to be quantitated must not exceed a ±15 percent difference when compared to the initial calibration curve. When this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis. If routine maintenance does not return the instrument performance to meet the QC requirements (Sec. 8.2) based on the last initial calibration, then a new initial calibration must be performed.

7.5.2.1 Analysts should use high and low concentrations of mixtures of single-component analytes and multi-component analytes for calibration verification.

7.5.3 Sample injection may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is recommended that standards be analyzed after every 10 (required after every 20 samples), and at the end of a set. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

7.5.3.1 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12 hr shift), or calibration standards interspersed within the samples. All samples that were injected after the standard that last met the QC criteria must be reinjected.

7.5.3.2 Although analysis of a single mid-concentration standard (standard mixture or multi-component analyte) will satisfy the minimum requirements, analysts are urged to use different calibration verification standards during organochlorine pesticide/PCB analyses. Also, multi-level standards (mixtures or multi-component analytes) are highly recommended to ensure that detector response remains stable for all analytes over the calibration range.

7.5.4 Establish absolute retention time windows for each analyte. Use the absolute retention time for each analyte from standards analyzed during that 12 hour shift as the midpoint of the window. The daily retention time window equals the midpoint ± three times the standard deviations.

7.5.4.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window.

7.5.4.2 Validation of gas chromatographic system qualitative performance: Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it.

7.5.5 Record the volume injected to the nearest 0.05 μL and the resulting peak size in area units. Using either the internal or the external calibration procedure (Method 8000), determine the identity and the quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes.

7.5.5.1 If the responses exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.5.5.2 If partially overlapping or coeluting peaks are found, change columns or try GC/MS quantitation, see Sec. 8 and Method 8270.

7.5.5.3 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

7.5.6 Identification of mixtures (i.e. PCBs and Toxaphene) is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and shape generated using either internal or external calibration procedures.

7.5.7 Quantitation of the target compounds is based on: 1) a reproducible response of the ECD or ELCD within the calibration range; and 2) a direct proportionality between the magnitude of response of the detector to peaks in the sample extract and the calibration standards. Proper quantitation requires the appropriate selection of a baseline from which the area or height of the characteristic peak(s) can be determined.

7.5.8 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to Method 3600 for the procedures to be followed in sample cleanup.

7.6 Quantitation of Multiple Component Analytes:

7.6.1 Multi-component analytes present problems in measurement. Suggestions are offered in the following sections for handing Toxaphene, Chlordane, PCBs, DDT, and BHC.

7.6.2 Toxaphene: Toxaphene is manufactured by the chlorination of camphenes, whereas Strobane results from the chlorination of a mixture of camphenes and pinenes. Quantitative calculation of Toxaphene or Strobane is difficult, but reasonable accuracy can be obtained. To calculate Toxaphene on GC/ECD: (a) adjust the sample size so that the major Toxaphene peaks are 10-70% of full-scale deflection (FSD); (b) inject a Toxaphene standard that is estimated to be within $\pm$10 ng of the sample; (c) quantitate using the five major peaks or the total area of the Toxaphene pattern.

7.6.2.1 To measure total area, construct the baseline of standard Toxaphene between its extremities; and construct the baseline under the sample, using the distances of the peak troughs to baseline on the standard as a guide. This procedure is made difficult by the fact that the relative heights and widths of the peaks in the sample will probably not be identical to the standard.

7.6.2.2 A series of Toxaphene residues have been calculated using the total peak area for comparison to the standard and also using the area of the last four peaks only, in both sample and standard. The agreement between the results obtained by the two methods justifies the use of the latter method for calculating Toxaphene in a sample where the early eluting portion of the Toxaphene chromatogram shows interferences from other substances such as DDT.

7.6.3 Chlordane is a technical mixture of at least 11 major components and 30 or more minor components. *Trans*- and *cis*-Chlordane (α and γ, respectively), are the two major components of technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch.

7.6.3.1 The GC pattern of a Chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of: constituents from the technical Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

7.6.3.2 Whenever possible, when a Chlordane residue does not resemble technical Chlordane, the analyst should quantitate the peaks of α-Chlordane, γ-Chlordane, and Heptachlor separately against the appropriate reference materials, and report the individual residues.

7.6.3.3 When the GC pattern of the residue resembles that of technical Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using the five major peaks or the total area. If the Heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation of the residue. If Heptachlor and/or Heptachlor epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected Chlordane area. (Note that octachloro epoxide, a metabolite of Chlordane, can easily be mistaken for Heptachlor epoxide on a nonpolar GC column.)

7.6.3.4 To measure the total area of the Chlordane chromatogram, inject an amount of technical Chlordane standard which will produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatograms.

7.6.4 Polychlorinated biphenyls (PCBs): Quantitation of residues of PCBs involves problems similar to those encountered in the quantitation of Toxaphene, Strobane, and Chlordane. In each case, the material is made up of numerous compounds which generate multi-peak chromatograms. Also, in each case, the chromatogram of the residue may not match that of the standard.

7.6.4.1 Mixtures of PCBs of various chlorine contents were sold for many years in the U.S. by the Monsanto Co. under the trade name Aroclor (1200 series and 1016). Although these Aroclors are no longer marketed, the PCBs remain in the environment and are sometimes found as residues in foods, especially fish. The Aroclors most commonly found in the environment are 1242, 1254, and 1260.

7.6.4.2 PCB residues are generally quantitated by comparison to the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

7.6.4.3 PCB Quantitation option #1- Quantitate the PCB residues by comparing the total area of the chlorinated biphenyl peaks to the total area of peaks from the appropriate Aroclor reference material. Measure the total area or height response from the common baseline under all the peaks. Use only those peaks from the sample that can be attributed to chlorobiphenyls. These peaks must also be present in the chromatogram of the reference materials. Option #1 should not be used if there are interference peaks within the Aroclor pattern, especially if they overlap PCB congeners.

7.6.4.4 PCB Quantitation option #2- Quantitate the PCB residues by comparing the responses of 3 to 5 major peaks in each appropriate Aroclor standard with the peaks obtained from the chlorinated biphenyls in the sample extract. The amount of Aroclor is calculated using an individual response factor for each of the major peaks. The results of the 3 to 5 determinations are averaged. Major peaks are defined as those peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. Late-eluting Aroclor peaks are generally the most stable in the environment.

7.6.4.5 When samples appear to contain weathered PCBs, treated PCBs, or mixtures of Aroclors, the use of Aroclor standards is not appropriate. Several diagnostic peaks useful for identifying non-Aroclor PCBs are given in Table 10. Analysts should examine chromatograms containing these peaks carefully, as these samples may contain PCBs. PCB concentrations may be estimated from specific congeners by adding the concentration of the congener peaks listed in Table 11. The congeners are analyzed as single components. This approach will provide reasonable accuracy for Aroclors 1016, 1232, 1242 and 1248 but will underestimate the concentrations of Aroclors 1254, 1260 and 1221. It is highly recommended that heavily weathered, treated, or mixed Aroclors be analyzed using GC/MS if concentration permits.

7.6.5 Hexachlorocyclohexane (BHC, from the former name, benzene hexachloride): Technical grade BHC is a cream-colored amorphous solid with a very characteristic musty odor; it consists of a mixture of six chemically distinct isomers and one or more heptachlorocyclohexanes and octachlorocyclohexanes. Commercial BHC preparations may show a wide variance in the percentage of individual isomers present. Quantitate each isomer (α, β, γ, and δ) separately against a standard of the respective pure isomer.

7.6.6 DDT: Technical DDT consists primarily of a mixture of 4,4'-DDT (approximately 75%) and 2,4'-DDT (approximately 25%). As DDT weathers, 4,4'-DDE, 2,4'-DDE, 4,4'-DDD, and 2,4'-DDD are formed. Since the 4,4'-isomers of DDT, DDE, and DDD predominate in the environment, these are the isomers normally regulated by US EPA and should be quantitated against standards of the respective pure isomer.

7.7 Suggested chromatography maintenance: Corrective measures may require any one or more of the following remedial actions.

7.7.1 Splitter connections: For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector (J&W Scientific or Restek), clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few inches (up to one foot) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

7.7.1.1 GC injector ports can be of critical concern, especially in the analysis of DDT and Endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation ("breakdown") of the analytes. Endrin and DDT breakdown to Endrin aldehyde, Endrin ketone, DDD, or DDE. When such breakdown is observed, clean and deactivate the injector port, break off at least 0.5 M of the column and remount it. Check the injector temperature and lower it to 205°C, if required. Endrin and DDT breakdown is less of a problem when ambient on-column injectors are used.

7.7.2 Metal injector body: Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

7.7.2.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone and then toluene; catch the rinsate in the beaker.

7.7.2.2 Prepare a solution of a deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, rinse the injector body with toluene, methanol, acetone, then hexane. Reassemble the injector and replace the columns.

7.7.3 Column rinsing: The column should be rinsed with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone; methylene chloride is a good final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control (QC) procedures including matrix spikes, duplicates and blanks. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If an extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

8.2 Quality control requirements for the GC system, including calibration and corrective actions, are found in Method 8000. The following steps are recommended as additional method QC.

8.2.1 The QC Reference Sample concentrate (Method 3500) should contain the organochlorine pesticides at 10 mg/L for water samples. If this method is to be used for analysis of Aroclors, Chlordane, or Toxaphene only, the QC Reference Sample should contain the most representative multi-component mixture at a concentration of 50 mg/L in acetone. The frequency of analysis of the QC reference sample analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. If the recovery of any compound found in the QC reference sample is less than 80 percent or greater than 120 percent of the certified value, the laboratory performance is judged to be out of control, and the problem must be corrected. A new set of calibration standards should be prepared and analyzed.

8.2.2 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in Method 8000).

If recovery is not within limits, the following are required:

8.2.2.1 Confirm that there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

8.2.2.2 Examine chromatograms for interfering peaks and for integrated areas.

8.2.2.3 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

8.2.2.4 Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

8.2.3 Include a calibration standard after each group of 20 samples (it is recommended that a calibration standard be included after every 10 samples to minimize the number of repeat injections) in the analysis sequence as a calibration check. The response factors for the calibration should be within 15 percent of the initial calibration. When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

8.2.4 Whenever quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different from the average area calculated during calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criteria must be reanalyzed.

8.3 DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

8.3.1 Calculate percent breakdown as follows:

$$\%\ \text{breakdown for 4,4'-DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD)}}{\text{peak areas (DDT + DDE + DDD)}} \times 100$$

$$\%\ \text{breakdown for Endrin} = \frac{\text{Total endrin degradation peak area (Endrin aldehyde + Endrin ketone)}}{\text{peak areas (Endrin + aldehyde + ketone)}} \times 100$$

8.3.2 The breakdown of DDT and Endrin should be measured before samples are analyzed and at the beginning of each 12 hour shift. Injector maintenance and recalibration should be completed if the breakdown is greater than 15% for either compound (Sec. 8.2.3).

8.4 GC/MS confirmation may be used for single column analysis. In addition, any compounds confirmed by two columns should also be confirmed by GC/MS if the concentration is sufficient for detection by GC/MS.

8.4.1 Full-scan GC/MS will normally require a minimum concentration near 10 ng/μL in the final extract for each single-component compound. Ion trap or selected ion monitoring will normally require a minimum concentration near 1 ng/μL.

8.4.2 The GC/MS must be calibrated for the specific target pesticides when it is used for quantitative analysis.

8.4.3 GC/MS may not be used for single column confirmation when concentrations are below 1 ng/μL.

8.4.4 GC/MS confirmation should be accomplished by analyzing the same extract used for GC/ECD analysis and the associated blank.

8.4.5 Use of the base/neutral-acid extract and associated blank may be used if the surrogates and internal standards do not interfere and it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral-acid extract even though the concentrations are high enough, a GC/MS analysis of the pesticide extract should be performed.

8.4.6 A QC reference sample of the compound must also be analyzed by GC/MS. The concentration of the QC reference standard must demonstrate the ability to confirm the pesticides/Aroclors identified by GC/ECD.

8.5 Whenever silica gel (Method 3630) or Florisil (Method 3620) cleanup is used, the analyst must demonstrate that the fractionation scheme is reproducible. Batch to batch variation in the composition of the silica gel material or overloading the column may cause a change in the distribution patterns of the organochlorine pesticides and PCBs. When compounds are found in two fractions, add the concentrations in the fractions, and corrections for any additional dilution.

9.0 METHOD PERFORMANCE

9.1 The MDL is defined in Chapter One. The MDL concentrations listed in Tables 1 and 2 were obtained using organic-free reagent water and sandy loam soil.

9.2 The chromatographic separations in this method have been tested in a single laboratory by using clean hexane and liquid and solid waste extracts that were spiked with the test compounds at three concentrations. Single-operator precision, overall precision, and method accuracy were found to be related to the concentration of the compound and the type of matrix.

9.3 This method has been applied in a variety of commercial laboratories for environmental and waste matrices. Performance data were obtained for a limited number of target analytes spiked into sewage sludge and dichloroethene still bottoms at high concentration levels. These data are provided in Tables 12 and 13.

9.4 The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.

9.5 Single laboratory accuracy data were obtained for organochlorine pesticides in a clay soil. The spiking concentration was 500 µg/kg. The spiking solution was mixed into the soil and then immediately transferred to the extraction device and immersed in the extraction solvent. The spiked sample was then extracted by Method 3541 (Automated Soxhlet). The data represent a single determination. Analysis was by capillary column gas chromatography/electron capture detector following Method 8081 for the organochlorine pesticides. These data are listed in Table 14 and were taken from Reference 14.

9.6 Single laboratory recovery data were obtained for PCBs in clay and soil. Oak Ridge National Laboratory spiked Aroclors 1254 and 1260 at concentrations of 5 and 50 ppm into portions of clay and soil samples and extracted these spiked samples using the procedure outlined in Method 3541. Multiple extractions using two different extractors were performed. The extracts were analyzed by Method 8081. The data are listed in Table 15 and were taken from Reference 15.

9.7 Multi-laboratory accuracy and precision data were obtained for PCBs in soil. Eight laboratories spiked Aroclors 1254 and 1260 into three portions of 10 g of Fuller's Earth on three non-consecutive days, followed by immediate extraction using Method 3541. Six of the laboratories spiked each Aroclor at 5 and 50 mg/kg and two laboratories spiked each Aroclor at 50 and 500 mg/kg. All extracts were analyzed by Oak Ridge National Laboratory, Oak Ridge, TN, using Method 8081. These data are listed in Table 16 and were taken from Reference 13.

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TABLE 1

GAS CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS FOR THE ORGANOCHLORINE PESTICIDES AND PCBs AS AROCLORS USING WIDE-BORE CAPILLARY COLUMNS SINGLE COLUMN METHOD OF ANALYSIS

Compound	Retention Time (min) DB 608[b]	DB 1701[b]	MDL[a] Water (µg/L)	MDL[a] Soil (µg/kg)
Aldrin	11.84	12.50	0.034	2.2
α-BHC	8.14	9.46	0.035	1.9
β-BHC	9.86	13.58	0.023	3.3
δ-BHC	11.20	14.39	0.024	1.1
γ-BHC (Lindane)	9.52	10.84	0.025	2.0
α-Chlordane	15.24	16.48	0.008	
γ-Chlordane	14.63	16.20	0.037	1.5
4,4'-DDD	18.43	19.56	0.050	4.2
4,4'-DDE	16.34	16.76	0.058	2.5
4,4'-DDT	19.48	20.10	0.081	3.6
Dieldrin	16.41	17.32	0.044	NA
Endosulfan I	15.25	15.96	0.030	2.1
Endosulfan II	18.45	19.72	0.040	2.4
Endosulfan Sulfate	20.21	22.36	0.035	3.6
Endrin	17.80	18.06	0.039	3.6
Endrin aldehyde	19.72	21.18	0.050	1.6
Heptachlor	10.66	11.56	0.040	2.0
Heptachlor epoxide	13.97	15.03	0.032	2.1
Methoxychlor	22.80	22.34	0.086	5.7
Toxaphene	MR	MR	NA	NA
Aroclor-1016	MR	MR	0.054	57.0
Aroclor-1221	MR	MR	NA	NA
Aroclor-1232	MR	MR	NA	NA
Aroclor-1242	MR	MR	NA	NA
Aroclor-1248	MR	MR	NA	NA
Aroclor-1254	MR	MR	NA	NA
Aroclor-1260	MR	MR	0.90	70.0

Water = Organic-free reagent water.
Soil = Sandy loam soil.
MR = Multiple peak responses.
NA = Data not available.

[a] MDL is the method detection limit. MDL was determined from the analysis of seven replicate aliquots of each matrix processed through the entire analytical method (extraction, silica gel cleanup, and GC/ECD analysis). MDL = t(n-1, 0.99) x SD, where t(n-1, 0.99) is the Student's t value appropriate for a 99% confidence interval and a standard deviation with n-1 degrees of freedom, and SD is the standard deviation of the seven replicate measurements.

[b] See Table 4 for GC operating conditions.

TABLE 2

GAS CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS FOR THE ORGANOCHLORINE PESTICIDES AND PCBs AS AROCLORS USING NARROW-BORE CAPILLARY COLUMNS SINGLE COLUMN METHOD OF ANALYSIS

Compound	Retention Time (min) DB 608[b]	DB 5[b]	MDL[a] Water (µg/L)	MDL[a] Soil (µg/kg)
Aldrin	14.51	14.70	0.034	2.2
α-BHC	11.43	10.94	0.035	1.9
β-BHC	12.59	11.51	0.023	3.3
δ-BHC	13.69	12.20	0.024	1.1
γ-BHC (Lindane)	12.46	11.71	0.025	2.0
α-Chlordane				
γ-Chlordane	17.34	17.02	0.037	1.5
4,4'-DDD	21.67	20.11	0.050	4.2
4,4'-DDE	19.09	18.30	0.058	2.5
4,4'-DDT	23.13	21.84	0.081	3.6
Dieldrin	19.67	18.74	0.044	NA
Endosulfan I	18.27	17.62	0.030	2.1
Endosulfan II	22.17	20.11	0.040	2.4
Endosulfan sulfate	24.45	21.84	0.035	3.6
Endrin	21.37	19.73	0.039	3.6
Endrin aldehyde	23.78	20.85	0.050	1.6
Heptachlor	13.41	13.59	0.040	2.0
Heptachlor epoxide	16.62	16.05	0.032	2.1
Methoxychlor	28.65	24.43	NA	NA
Toxaphene	MR	MR	0.086	5.7
Aroclor-1016	MR	MR	NA	NA
Aroclor-1221	MR	MR	0.054	57.0
Aroclor-1232	MR	MR	NA	NA
Aroclor-1242	MR	MR	NA	NA
Aroclor-1248	MR	MR	NA	NA
Aroclor-1254	MR	MR	NA	NA
Aroclor-1260	MR	MR	0.90	70.0

Water = Organic-free reagent water.
Soil = Sandy loam soil.
MR = Multiple peak responses.
NA = Data not available.

TABLE 2
(Continued)

[a] MDL is the method detection limit. MDL was determined from the analysis of seven replicate aliquots of each matrix processed through the entire analytical method (extraction, cleanup, and GC/ECD analysis). MDL = t(n-1, 0.99) x SD, where t(n-1, 0.99) is the Student's t value appropriate for a 99% confidence interval and a standard deviation with n-1 degrees of freedom, and SD is the standard deviation of the seven replicate measurements.

[b] 30 m x 0.25 mm ID DB-608 1 μm film thickness, see Table 4 for GC operating conditions.

[c] 30 m x 0.25 mm ID DB-5 1 μm film thickness, see Table 4 for GC operating conditions.

TABLE 3

DETERMINATION OF ESTIMATED QUANTITATION LIMITS (EQLs) FOR VARIOUS MATRICES*

Matrix	Factor
Ground water	10
Low-concentration soil by sonication with GPC cleanup	670
High-concentration soil and sludges by sonication	10,000
Non-water miscible waste	100,000

* EQL = [Method detection limit for water (see Table 1 or Table 2) wide-bore or narrow-bore options] x [Factor found in this table]. For nonaqueous samples, the factor is on a wet-weight basis. Sample EQLs are highly matrix-dependent. The EQLs to be determined herein are provided for guidance and may not always be achievable.

TABLE 4
GC OPERATING CONDITIONS FOR ORGANOCHLORINE COMPOUNDS
SINGLE COLUMN ANALYSIS

Narrow-bore columns:

Narrow-bore Column 1 - 30 m x 0.25 or 0.32 mm internal diameter (ID) fused silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1 μm film thickness.

Carrier gas (He)	16 psi
Injector temperature	225°C
Detector temperature	300°C
Initial temperature	100°C, hold 2 minutes
Temperature program	100°C to 160°C at 15°C/min, followed by 160°C to 270°C at 5°C/min
Final temperature	270°C

Narrow-bore Column 2 - 30 m x 0.25 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, or equivalent), 25 μm coating thickness, 1 μm film thickness

Carrier gas (N_2)	20 psi
Injector temperature	225°C
Detector temperature	300°C
Initial temperature	160°C, hold 2 minutes
Temperature program	160°C to 290°C at 5°C/min
Final temperature	290°C, hold 1 min

Wide-bore columns:

Wide-bore Column 1 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB-608, SPB-608, RTx-35, or equivalent), 0.5 μm or 0.83 μm film thickness.

Wide-bore Column 2 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent), 1.0 μm film thickness.

Carrier gas (He)	5-7 mL/minute
Makeup gas	
argon/methane (P-5 or P-10) or N_2	30 mL/min
Injector temperature	250°C
Detector temperature	290°C
Initial temperature	150°C, hold 0.5 minute
Temperature program	150°C to 270°C at 5°C/min
Final temperature	270°C, hold 10 min

(continued)

TABLE 4 (Continued)
GC OPERATING CONDITIONS FOR ORGANOCHLORINE COMPOUNDS
SINGLE COLUMN ANALYSIS

Wide-bore Columns (continued)

Wide-bore Column 3 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 1.5 μm film thickness.

Carrier gas (He)	6 mL/minute
Makeup gas argon/methane (P-5 or P-10) or N_2	30 mL/min
Injector temperature	205°C
Detector temperature	290°C
Initial temperature	140°C, hold 2 min
Temperature program	140°C to 240°C at 10°C/min, hold 5 minutes at 240°C, 240°C to 265°C at 5°C/min
Final temperature	265°C, hold 18 min

TABLE 5
RETENTION TIMES OF THE ORGANOCHLORINE PESTICIDES*
DUAL COLUMN METHOD OF ANALYSIS

Compound	CAS No.	DB-5 RT(min)	DB-1701 RT(min)
DBCP	96-12-8	2.14	2.84
Hexachlorocyclopentadiene	77-47-4	4.49	4.88
Etridiazole	2593-15-9	6.38	8.42
Chloroneb	2675-77-6	7.46	10.60
Hexachlorobenzene	118-74-1	12.79	14.58
Diallate	2303-16-4	12.35	15.07
Propachlor	1918-16-17	9.96	15.43
Trifluralin	1582-09-8	11.87	16.26
α-BHC	319-84-6	12.35	17.42
PCNB	82-68-8	14.47	18.20
γ-BHC	58-89-9	14.14	20.00
Heptachlor	76-44-8	18.34	21.16
Aldrin	309-00-2	20.37	22.78
Alachlor	15972-60-8	18.58	24.18
Chlorothalonil	1897-45-6	15.81	24.42
Alachlor	15972-60-8	18.58	24.18
β-BHC	319-85-7	13.80	25.04
Isodrin	465-73-6	22.08	25.29
DCPA	1861-32-1	21.38	26.11
δ-BHC	319-86-8	15.49	26.37
Heptachlor epoxide	1024-57-3	22.83	27.31
Endosulfan-I	959-98-8	25.00	28.88
γ-Chlordane	5103-74-2	24.29	29.32
α-Chlordane	5103-71-9	25.25	29.82
trans-Nonachlor	39765-80-5	25.58	30.01
4,4'-DDE	72-55-9	26.80	30.40
Dieldrin	60-57-1	26.60	31.20
Captan	133-06-2	23.29	31.47
Perthane	72-56-0	28.45	32.18
Endrin	72-20-8	27.86	32.44
Chloropropylate	99516-95-7	28.92	34.14
Chlorobenzilate	510-15-6	28.92	34.42
Nitrofen	1836-75-5	27.86	34.42
4,4'-DDD	72-54-8	29.32	35.32
Endosulfan II	33213-65-9	28.45	35.51
4,4'-DDT	50-29-3	31.62	36.30
Endrin aldehyde	7421-93-4	29.63	38.08
Mirex	2385-85-5	37.15	38.79
Endosulfan sulfate	1031-07-8	31.62	40.05

continued

TABLE 5
(Continued)

Compound	CAS No.	DB-5 RT(min)	DB-1701 RT(min)
Methoxychlor	72-43-5	35.33	40.31
Captafol	2425-06-1	32.65	41.42
Endrin ketone	53494-70-5	33.79	42.26
trans-Permethrin	51877-74-8	41.50	45.81
Kepone	143-50-0	31.10	b
Dicofol	115-32-2	35.33	b
Dichlone	117-80-6	15.17	b
α,α'-Dibromo-m-xylene		9.17	11.51
2-Bromobiphenyl		8.54	12.49

[a]The GC operating conditions were as follows: 30-m x 0.53-mm ID DB-5 (0.83-μm film thickness) and 30-m x 0.53-mm ID DB-1701 (1.0-μm film thickness) connected to an 8-in injection tee (Supelco Inc.). Temperature program: 140°C (2-min hold) to 270°C (1-min hold) at 2.8°C/min; injector temperature 250°C; detector temperature 320°C; helium carrier gas 6 mL/min; nitrogen makeup gas 20 mL/min.
[b]Not detected at 2 ng per injection.

TABLE 6
GC OPERATING CONDITIONS FOR ORGANOCHLORINE PESTICIDES
FOR DUAL COLUMN METHOD OF ANALYSIS
LOW TEMPERATURE, THIN FILM

Column 1:

Type: DB-1701 (J&W) or equivalent
Dimensions: 30 m x 0.53 mm ID
Film Thickness (μm): 1.0

Column 2:

Type: DB-5 (J&W) or equivalent
Dimensions: 30 m x 0.53 mm ID
Film Thickness (μm): 0.83

Carrier gas flowrate (mL/min): 6 (Helium)

Makeup gas flowrate (mL/min): 20 (Nitrogen)

Temperature program: 140°C (2 min hold) to 270°C (1 min hold) at 2.8°C/min

Injector temperature: 250°C

Detector temperature: 320°C

Injection volume: 2 μL

Solvent: Hexane

Type of injector: Flash vaporization

Detector type: Dual ECD

Range: 10

Attenuation: 64 (DB-1701)/32 (DB-5)

Type of splitter: Supelco 8 in injection tee

TABLE 7
GC OPERATING CONDITIONS FOR ORGANOCHLORINE PESTICIDES
FOR THE DUAL COLUMN METHOD OF ANALYSIS
HIGH TEMPERATURE, THICK FILM

Column 1:

Type: DB-1701 (J&W) or equivalent
Dimensions: 30 m x 0.53 mm ID
Film Thickness: 1.0 μm

Column 2:

Type: DB-5 (J&W) or equivalent
Dimensions: 30 m x 0.53 mm ID
Film Thickness: 1.5 μm

Carrier gas flowrate (mL/min): 6 (Helium)

Makeup gas flowrate (mL/min): 20 (Nitrogen)

Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Injector temperature: 250°C

Detector temperature: 320°C

Injection volume: 2 μL

Solvent: Hexane

Type of injector: Flash vaporization

Detector type: Dual ECD

Range: 10

Attenuation: 64 (DB-1701)/64 (DB-5)

Type of splitter: J&W Scientific press-fit Y-shaped inlet splitter

TABLE 8 SUMMARY OF RETENTION TIMES (MIN) OF AROCLORS ON THE DB-5 COLUMN[a] DUAL SYSTEM OF ANALYSIS

Peak No.[b]	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Pesticide eluting at same retention time
1		5.85	5.85					
2		7.63	7.64	7.57				
3	8.41	8.43	8.43	8.37				
4	8.77	8.77	8.78	8.73				
5	8.98	8.99	9.00	8.94	8.95			
6	9.71			9.66				
7	10.49	10.50	10.50	10.44	10.45			
8	10.58	10.59	10.59	10.53				
9	10.90		10.91	10.86	10.85			
10	11.23	11.24	11.24	11.18	11.18			Chlorothalonil (11.18)
11	11.88		11.90	11.84	11.85			
12	11.99		12.00	11.95				
13	12.27	12.29	12.29	12.24	12.24			
14	12.66	12.68	12.69	12.64	12.64			
15	12.98	12.99	13.00	12.95	12.95			
16	13.18		13.19	13.14	13.15			
17	13.61		13.63	13.58	13.58	13.59	13.59	
18	13.80		13.82	13.77	13.77	13.78		
19	13.96		13.97	13.93	13.93	13.90		
20	14.48		14.50	14.46	14.45	14.46		
21	14.63		14.64	14.60	14.60			
22	14.99		15.02	14.98	14.97	14.98		
23	15.35		15.36	15.32	15.31	15.32		
24	16.01			15.96				
25			16.14	16.08	16.08	16.10		
26	16.27		16.29	16.26	16.24	16.25	16.26	Captan (16.21)
27						16.53		
28			17.04		16.99	16.96	16.97	gamma-Chlordane (16.95)
29			17.22	17.19	17.19	17.19	17.21	
30			17.46	17.43	17.43	17.44		
31					17.69	17.69		
32				17.92	17.91	17.91		
33				18.16	18.14	18.14		
34			18.41	18.37	18.36	18.36	18.37	4,4'-DDE (18.38)
35			18.58	18.56	18.55	18.55		Dieldrin (18.59)
36							18.68	
37			18.83	18.80	18.78	18.78	18.79	
38			19.33	19.30	19.29	19.29	19.29	
39						19.48	19.48	
40						19.81	19.80	
41			20.03	19.97	19.92	19.92		Chloropropylate (19.91) Endosulfan II (19.91)
42						20.28	20.28	
43				20.46	20.45			
44						20.57	20.57	
45				20.85	20.83	20.83	20.83	
46			21.18	21.14	21.12	20.98		Kepone (20.99)
47					21.36	21.38	21.38	
48						21.78	21.78	4,4'-DDT (21.75) Endosulfan sulfate (21.75)
49				22.08	22.05	22.04	22.03	
50						22.38	22.37	
51						22.74	22.73	Captafol (22.71)
52						22.96	22.95	
53						23.23	23.23	
54							23.42	
55						23.75	23.73	Endrin ketone (23.73)

(continued)

[a]The GC operating conditions are given in Table 7.

TABLE 8 CONTINUED

Peak No.	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Pesticide eluting at same retention time
56						23.99	23.97	
57							24.16	
58						24.27		Methoxychlor (24.29) Dicofol (24.29)
59							24.45	
60						24.61	24.62	
61						24.93	24.91	
62							25.44	
63						26.22	26.19	Mirex (26.19)
64							26.52	
65							26.75	
66							27.41	
67							28.07	
68							28.35	
69							29.00	

[a]The GC operating conditions are given in Table 7.
[b]These are sequentially numbered from elution order and are not isomer numbers

TABLE 9 SUMMARY OF RETENTION TIMES (MIN) OF AROCLORS ON THE DB-1701 COLUMN[a] DUAL SYSTEM OF ANALYSIS

Peak No.[b]	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Pesticide eluting at same retention time
1		4.45	4.45					
2		5.38						
3		5.78						
4		5.86	5.86					
5	6.33	6.34	6.34	6.28				
6	6.78	6.78	6.79	6.72				
7	6.96	6.96	6.96	6.90	6.91			Trifluralin (6.96)
8	7.64			7.59				
9	8.23	8.23	8.23	8.15	8.16			
10	8.62	8.63	8.63	8.57				
11	8.88		8.89	8.83	8.83			
12	9.05	9.06	9.06	8.99	8.99			
13	9.46		9.47	9.40	9.41			
14	9.77	9.79	9.78	9.71	9.71			
15	10.27	10.29	10.29	10.21	10.21			
16	10.64	10.65	10.66	10.59	10.59			
17				10.96	10.95	10.95		
18	11.01		11.02	11.02	11.03			
19	11.09		11.10					
20	11.98		11.99	11.94	11.93	11.93		
21	12.39		12.39	12.33	12.33	12.33		
22			12.77	12.71	12.69			
23	12.92			12.94	12.93			
24	12.99		13.00	13.09	13.09	13.10		
25	13.14		13.16					
26						13.24		
27	13.49		13.49	13.44	13.44			
28	13.58		13.61	13.54	13.54	13.51	13.52	
29				13.67		13.68		
30			14.08	14.03	14.03	14.03	14.02	
31			14.30	14.26	14.24	14.24	14.25	
32					14.39	14.36		
33			14.49	14.46	14.46			
34						14.56	14.56	
35					15.10	15.10		
36			15.38	15.33	15.32	15.32		Chlordane (15.32)
37			15.65	15.62	15.62	15.61	16.61	4,4'-DDE (15.67)
38			15.78	15.74	15.74	15.74	15.79	
39			16.13	16.10	16.10	16.08		
40							16.19	
41						16.34	16.34	
42						16.44	16.45	
43						16.55		
44			16.77	16.73	16.74	16.77	16.77	Perthane (16.71)
45			17.13	17.09	17.07	17.07	17.08	
46						17.29	17.31	
47				17.46	17.44	17.43	17.43	
48				17.69	17.69	17.68	17.68	
49					18.19	18.17	18.18	
50				18.48	18.49	18.42	18.40	
51						18.59		
52						18.86	18.86	
53				19.13	19.13	19.10	19.09	Endosulfan II (19.05)
54						19.42	19.43	

(continued)

[a]The GC operating conditions are given in Table 7.

TABLE 9 CONTINUED

Peak No.	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Pesticide eluting at same retention time
55						19.55	19.59	4,4'-DDT (19.54)
56						20.20	20.21	
57						20.34		
58							20.43	
59					20.57	20.55		
60						20.62	20.66	Endrin aldehyde (20.69)
61						20.88	20.87	
62							21.03	
63						21.53	21.53	
64						21.83	21.81	
65						23.31	23.27	
66							23.85	
67							24.11	
68							24.46	
69							24.59	
70							24.87	
71							25.85	
72							27.05	
73							27.72	

[a]The GC operating conditions are given in Table 7.
[b]These are sequentially numbered from elution order and are not isomer numbers

TABLE 10
PEAKS DIAGNOSTIC OF PCBs OBSERVED IN 0.53 mm ID COLUMN
SINGLE COLUMN ANALYSIS

Peak No.[c]	RT on DB 608[a]	RT on DB 1701[a]	Aroclor[b]	Elution Order
I	4.90	4.66	1221	Before TCmX
II	7.15	6.96	1221, 1232, 1248	Before α-BHC
III	7.89	7.65	1061, 1221, 1232, 1242,	Before α-BHC
IV	9.38	9.00	1016, 1232, 1242, 1248,	just after α-BHC on DB-1701;just before γ-BHC on DB-608
V	10.69	10.54	1016, 1232, 1242,	1248 α-BHC and heptachlor on DB-1701; just after heptachlor on DB-608
VI	14.24	14.12	1248, 1254	γ-BHC and heptachlor epoxide on DB-1701; heptachlor epoxide and γ-Chlordane on DB-608
VII	14.81	14.77	1254	Heptachlor epoxide and γ-Chlordane on DB-1701; α- and γ-Chlordane on DB-608
VIII	16.71	16.38	1254	DDE and Dieldrin on DB-1701; Dieldrin and Endrin on DB-608
IX	19.27	18.95	1254, 1260	Endosulfan II on DB-1701; DDT on DB-608

Continued

TABLE 10 (Continued)
PEAKS DIAGNOSTIC OF PCBs OBSERVED IN 0.53 mm ID COLUMN
SINGLE COLUMN ANALYSIS

Peak No.	RT on DB 608[a]	RT on DB 1701[a]	Aroclor[b]	Elution Order
X	21.22	21.23	<u>1260</u>	Endrin aldehyde and Endosulfan sulfate on DB-1701; Endosulfan sulfate and Methoxychlor on on DB-608
XI	22.89	22.46	1260	Just before endrin ketone on DB-1701; after endrin ketone on DB-608

[a] Temperature program: T_i = 150°C, hold 30 seconds; increase temperature at 5°C/minutes to 275°C.

[b] Underlined Aroclor indicates the largest peak in the pattern.

[c] These are sequentially numbered from elution order and are not isomer numbers

TABLE 11 SPECIFIC PCB CONGENERS IN AROCLORS

Congener	IUPAC number	Aroclor 1016	1221	1232	1242	1248	1254	1260
Biphenyl	--		X					
2CB	1	X	X	X	X			
23DCB	5	X	X	X	X	X		
34DCB	12	X		X	X	X		
244'TCB	28*	X		X	X	X	X	
22'35'TCB	44			X	X	X	X	
23'44'TCB	66*					X	X	X
233'4'6PCB	110						X	
23'44'5PCB	118*						X	X
22'44'55'HCB	153							X
22'344'5'HCB	138							X
22'344'55'HpCB	180							X
22'33'44'5HpCB	170							X

*apparent co-elution of two major peaks:

28 with 31 (2,4',5 trichloro)
66 with 95 (2,2',3,5',6 pentachloro)
118 with 149 (2,2',3,4',5',6 hexachloro)

TABLE 12 ANALYTE RECOVERY FROM SEWAGE SLUDGE

Compound	Sonication		Soxhlet	
	%Recovery	%RSD	%Recovery	%RSD
Hexachloroethane	80	7	79	1
2-Chloronapthalene	50	56	67	8
4-Bromodiphenyl ether	118	14	nd	
α-BHC	88	25	265	18
γ-BHC	55	9	155	29
Heptachlor	60	13	469	294
Aldrin	92	33	875	734
β-BHC	351	71	150	260
δ-BHC	51	11	57	2
Heptachlor epoxide	54	11	70	3
Endosulfan I	52	11	70	4
γ-Chlordane	50	9	65	1
α-Chlordane	49	8	66	0
DDE	52	11	74	1
Dieldrin	89	19	327	7
Endrin	56	10	92	15
Endosulfan II	52	10	88	11
DDT	57	10	95	17
Endrin aldehyde	45	6	42	10
DDD	57	11	99	8
Tetrachloro-m-xylene	71	19	82	1
Decachlorobiphenyl	26	23	28	48

Concentration spiked in the sample: 500-1000 ng/g
Three replicates/sample

Extraction solvent, Method 3540 - methylene chloride
Extraction solvent, Method 3550 - methylene chloride/acetone (1:1)

Cleanup - Method 3640

GC column - DB-608, 30M X 0.53 mm ID

TABLE 13 ANALYTE RECOVERY FROM DCE STILL BOTTOMS

Compound	Sonication		Soxhlet	
	%Recovery	%RSD	%Recovery	%RSD
Hexachloroethane	70	2	50	30
2-Chloronapthalene	59	3	35	35
4-Bromodiphenyl ether	159	14	128	137
α-BHC	55	7	47	25
β-BHC	43	6	30	30
Heptachlor	48	6	55	18
Aldrin	48	5	200	258
β-BHC	51	7	75	42
δ-BHC	43	4	119	129
Heptachlor epoxide	47	6	66	34
Endosulfan I	47	4	41	18
γ-Chlordane	48	5	47	13
α-Chlordane	45	5	37	21
DDE	45	4	70	40
Dieldrin	45	5	58	24
Endrin	50	6	41	23
Endosulfan II	49	5	46	17
DDT	49	4	40	29
Endrin aldehyde	40	4	29	20
DDD	48	5	35	21
Tetrachloro-m-xylene	49	2	176	211
Decachlorobiphenyl	17	29	104	93

Concentration spiked in the sample: 500-1000 ng/g
Three replicates/sample

Extraction solvent, Method 3540 - methylene chloride
Extraction solvent, Method 3550 - methylene chloride/acetone (1:1)

Cleanup - Method 3640

GC column - DB-608, 30M X 0.53 mm ID

TABLE 14
SINGLE LABORATORY ACCURACY DATA FOR THE EXTRACTION OF ORGANOCHLORINE PESTICIDES FROM SPIKED CLAY SOIL BY METHOD 3541 (AUTOMATED SOXHLET)[a]

Compound Name	Spike Level µg/kg	% Recovery DB-5	% Recovery DB-1701
α-BHC	500	89	94
β-BHC	500	86	b
Heptachlor	500	94	95
Aldrin	500	b	92
Heptachlor epoxide	500	97	97
trans-Chlordane	500	94	95
Endosulfan I	500	92	92
Dieldrin	500	b	113
Endrin	500	111	104
Endosulfan II	500	104	104
4,4'-DDT	500	b	b
Mirex	500	108	102

a The operating conditions for the automated Soxhlet were as follows: immersion time 45 min; extraction time 45 min; the sample size was 10 g clay soil, extraction solvent, 1:1 acetone/hexane. No equilibration time following spiking.

b Not able to determine because of interference.

Data taken from Reference 14.

TABLE 15
SINGLE LABORATORY RECOVERY DATA FOR EXTRACTION OF
PCBS FROM CLAY AND SOIL BY METHOD 3541[a] (AUTOMATED SOXHLET)

Matrix	Compound	Spike Level (ppm)	Trial	Percent Recovery[b]
Clay	Aroclor-1254	5	1	87.0
			2	92.7
			3	93.8
			4	98.6
			5	79.4
			6	28.3
Clay	Aroclor-1254	50	1	65.3
			2	72.6
			3	97.2
			4	79.6
			5	49.8
			6	59.1
Clay	Aroclor-1260	5	1	87.3
			2	74.6
			3	60.8
			4	93.8
			5	96.9
			6	113.1
Clay	Aroclor-1260	50	1	73.5
			2	70.1
			3	92.4
			4	88.9
			5	90.2
			6	67.3
Soil	Aroclor-1254	5	1	69.7
			2	89.1
			3	91.8
			4	83.2
			5	62.5
Soil	Aroclor-1254	50	1	84.0
			2	77.5
			3	91.8
			4	66.5
			5	82.3
			6	61.6

(continued)

TABLE 15
(continued)

Matrix	Compound	Spike Level (ppm)	Trial	Percent Recovery[b]
Soil	Aroclor-1260	5	1	83.9
			2	82.8
			3	81.6
			4	96.2
			5	93.7
			6	93.8
			7	97.5
Soil	Aroclor-1260	50	1	76.9
			2	69.4
			3	92.6
			4	81.6
			5	83.1
			6	76.0

a The operating conditions for the automated Soxhlet were as follows: immersion time 60 min; reflux time 60 min.

b Multiple results from two different extractors.

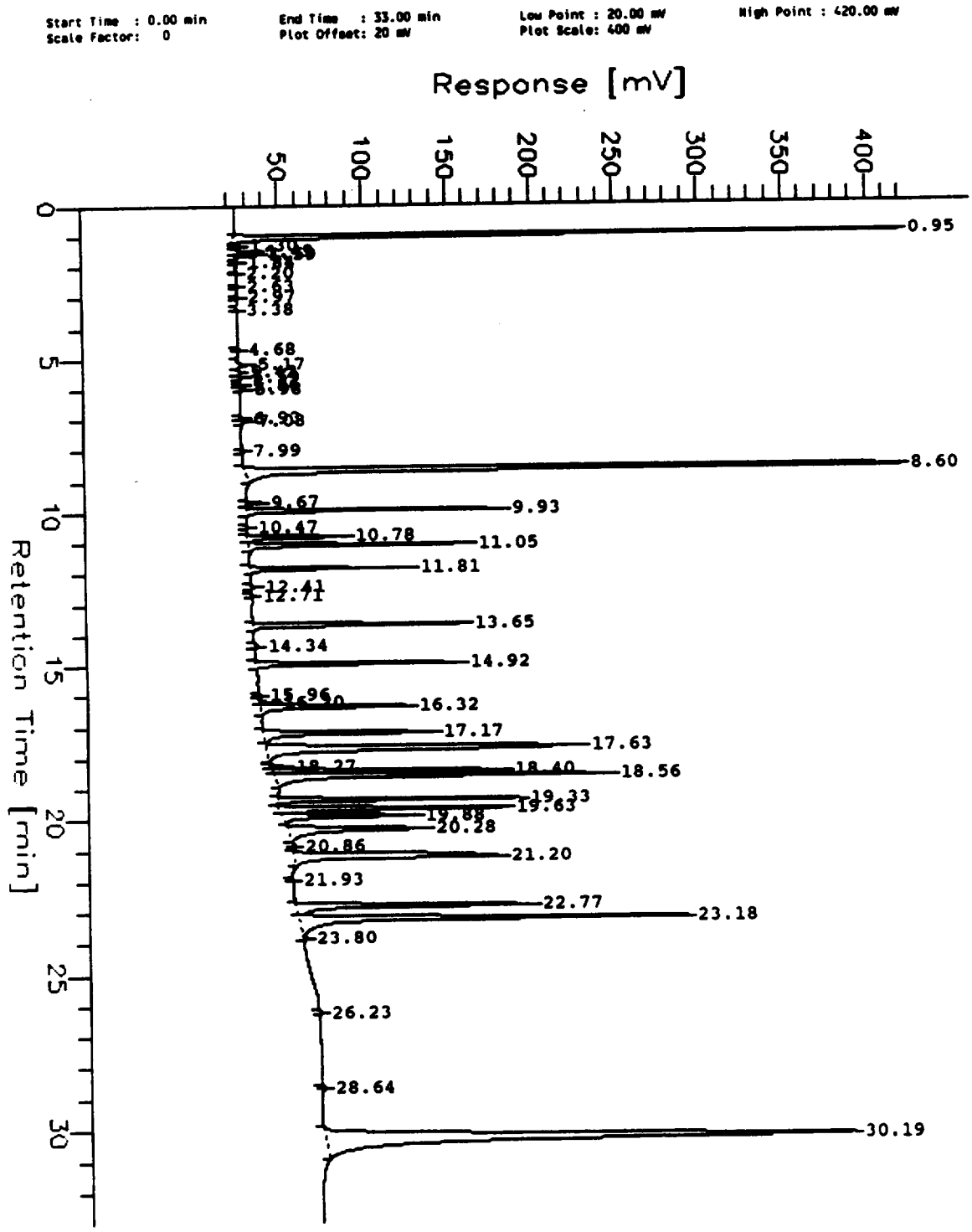
Data from Reference 15.

TABLE 16. MULTI-LABORATORY PRECISION AND ACCURACY DATA FOR THE EXTRACTION OF PCBS FROM SPIKED SOIL BY METHOD 3541 (AUTOMATED SOXHLET)

Laboratory		PCB Percent Recovery Aroclor 1254 PCB Level 5	50	500	1260 PCB Level 5	50	500	All Levels
Lab 1	Num	3.0	3.0		3.0	3.0		12.0
	Average	101.2	74.0		83.9	78.5		84.4
	St Dev	34.9	41.8		7.4	7.4		26.0
Lab 2	Num		6.0	6.0		6.0	6.0	24.0
	Average		56.5	66.9		70.1	74.5	67.0
	St Dev		7.0	15.4		14.5	10.3	13.3
Lab 3	Num	3.0	3.0		3.0	3.0		12.0
	Average	72.8	63.3		70.6	57.2		66.0
	St Dev	10.8	8.3		2.5	5.6		9.1
Lab 4	Num	6.0	6.0		6.0	6.0		24.0
	Average	112.6	144.3		100.3	84.8		110.5
	St Dev	18.2	30.4		13.3	3.8		28.5
Lab 5	Num		3.0	3.0		3.0	3.0	12.0
	Average		97.1	80.1		79.5	77.0	83.5
	St Dev		8.7	5.1		3.1	9.4	10.3
Lab 6	Num	2.0	3.0		3.0	4.0		12.0
	Average	140.9	127.7		138.7	105.9		125.4
	St Dev	4.3	15.5		15.5	7.9		18.4
Lab 7	Num	3.0	3.0		3.0	3.0		12.0
	Average	100.1	123.4		82.1	94.1		99.9
	St Dev	17.9	14.6		7.9	5.2		19.0
Lab 8	Num	3.0	3.0		3.0	3.0		12.0
	Average	65.0	38.3		92.8	51.9		62.0
	St Dev	16.0	21.9		36.5	12.8		29.1
All Laboratories	Num	20.0	30.0	9.0	21.0	31.0	9.0	120.0
	Average	98.8	92.5	71.3	95.5	78.6	75.3	87.6
	St Dev	28.7	42.9	14.1	25.3	18.0	9.5	29.7

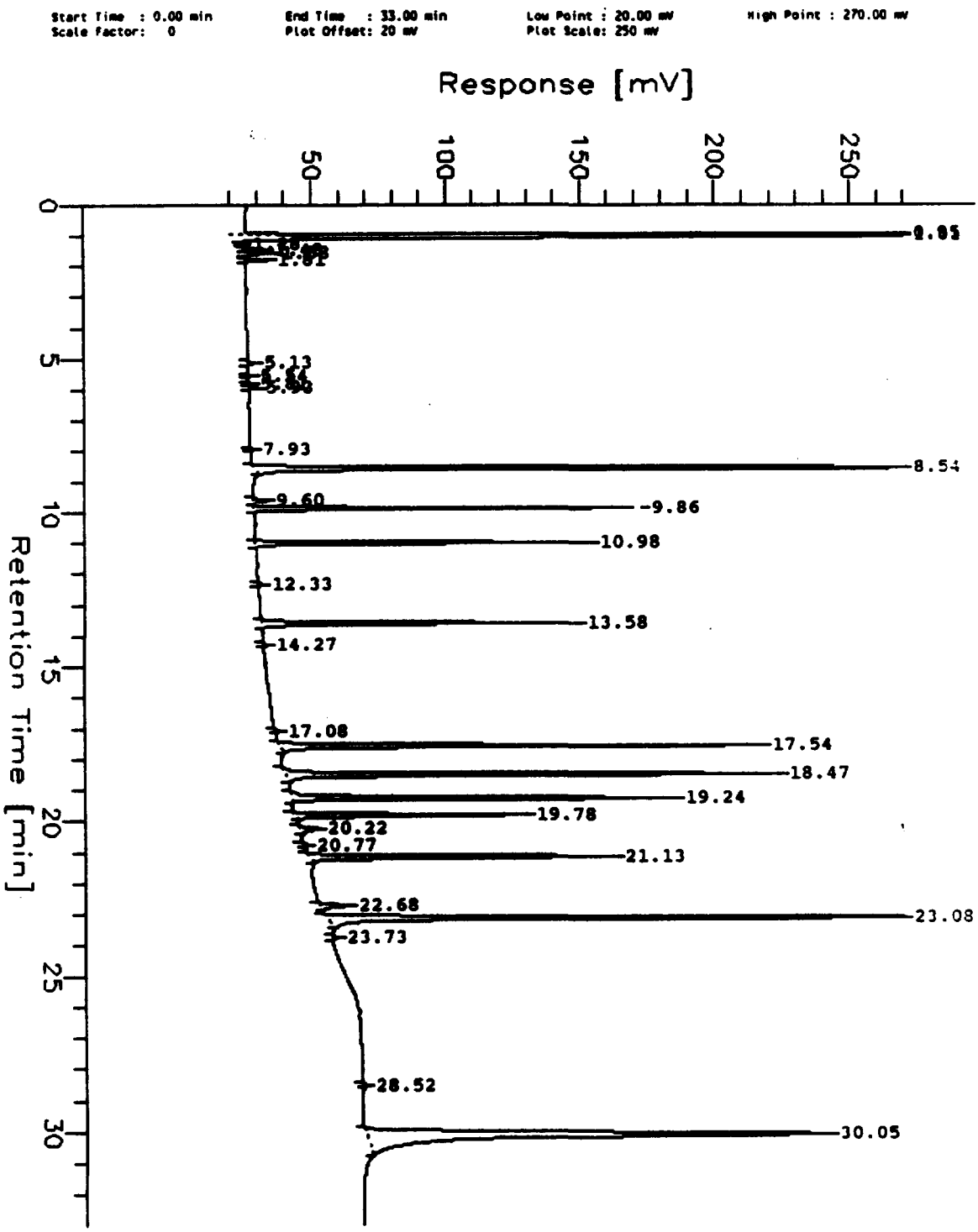
Data from Reference 13.

FIGURE 1.
GAS CHROMATOGRAM OF THE MIXED ORGANOCHLORINE PESTICIDE STANDARD



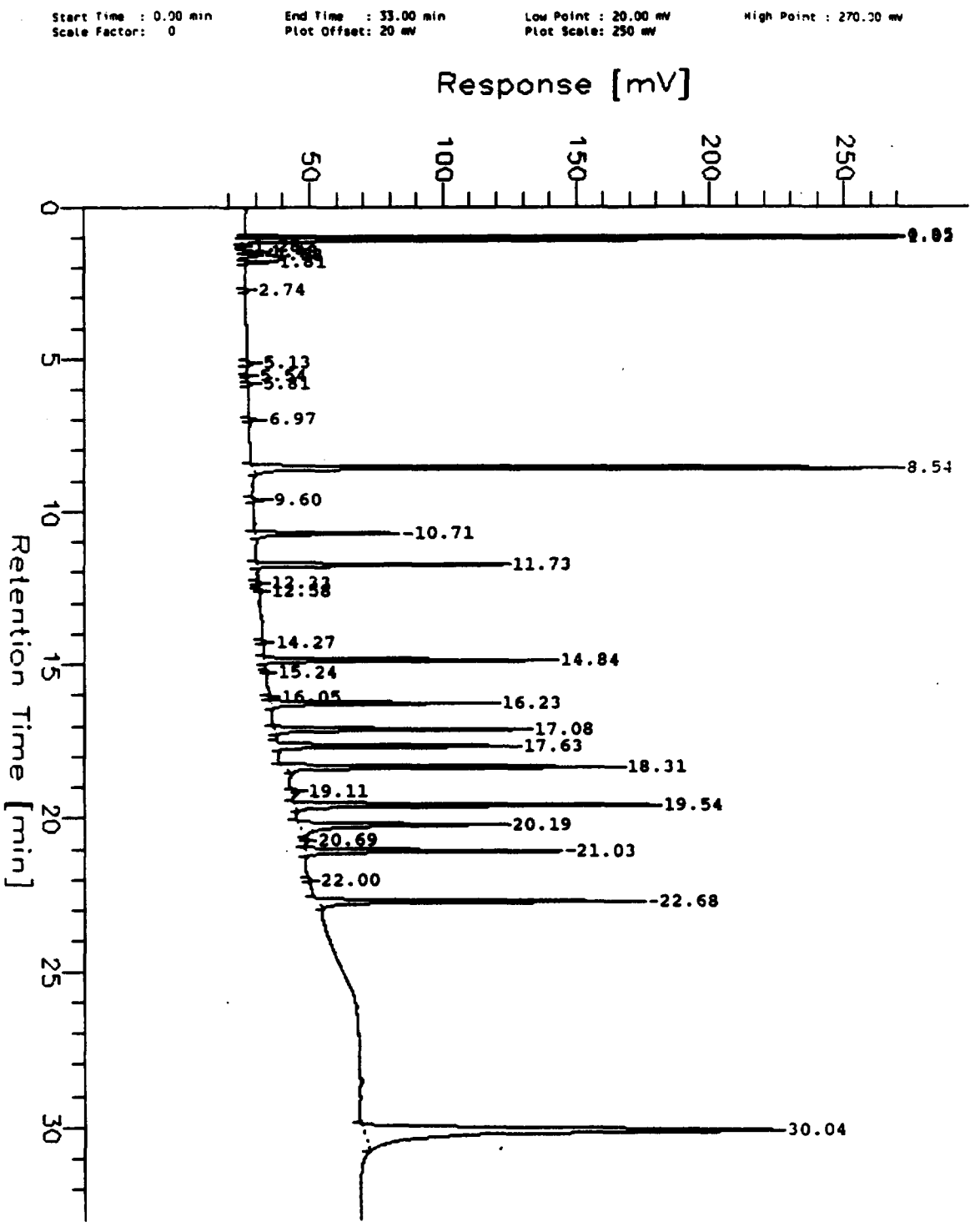
Column: 30 m x 0.25 mm ID, DB-5
Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C; carrier He at 16 psi.

FIGURE 2.
GAS CHROMATOGRAM OF INDIVIDUAL ORGANOCHLORINE PESTICIDE STANDARD MIX A



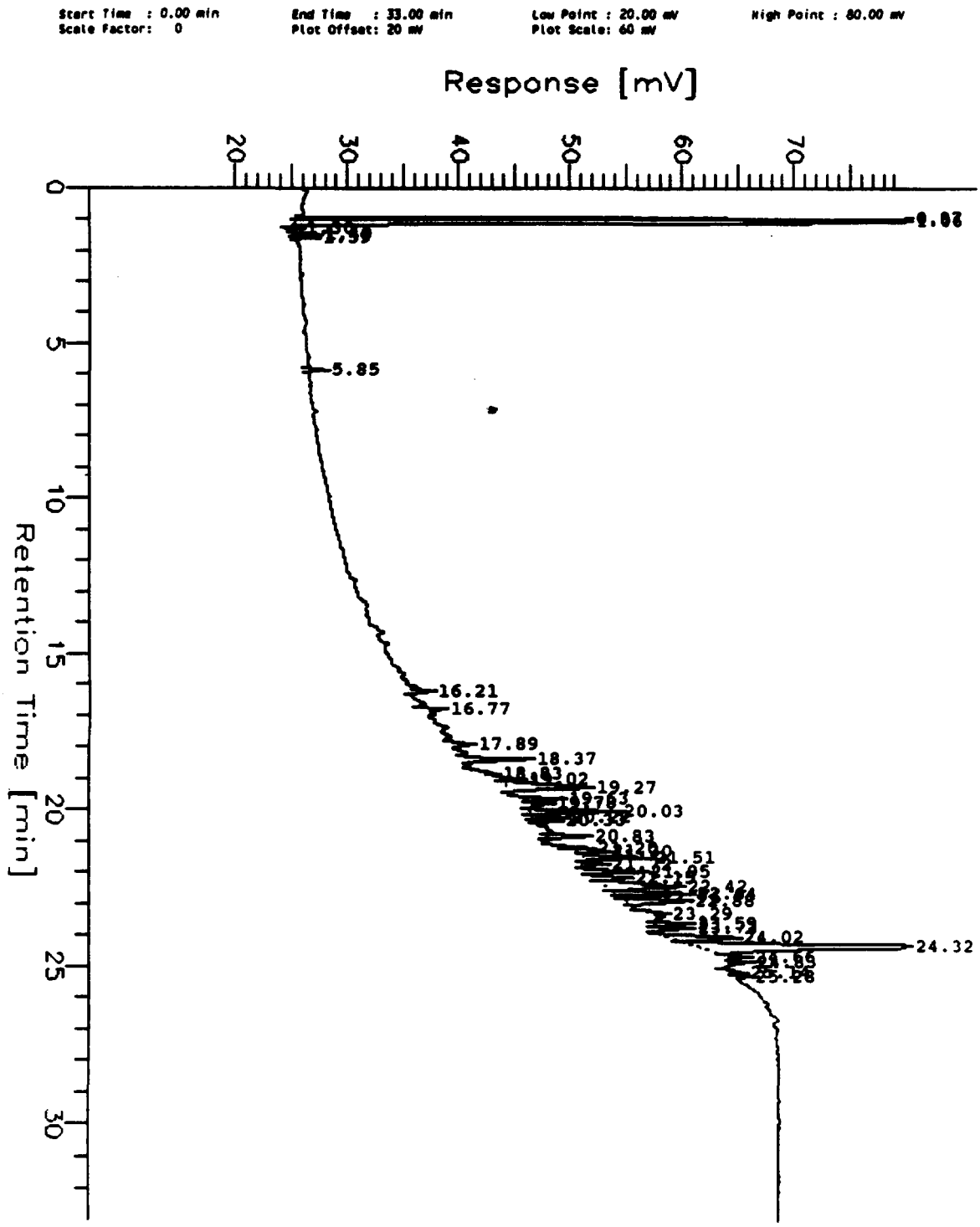
Column: 30 m x 0.25 mm ID, DB-5
Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C; carrier He at 16 psi.

FIGURE 3
GAS CHROMATOGRAM OF INDIVIDUAL ORGANOCHLORINE PESTICIDE STANDARD MIX B



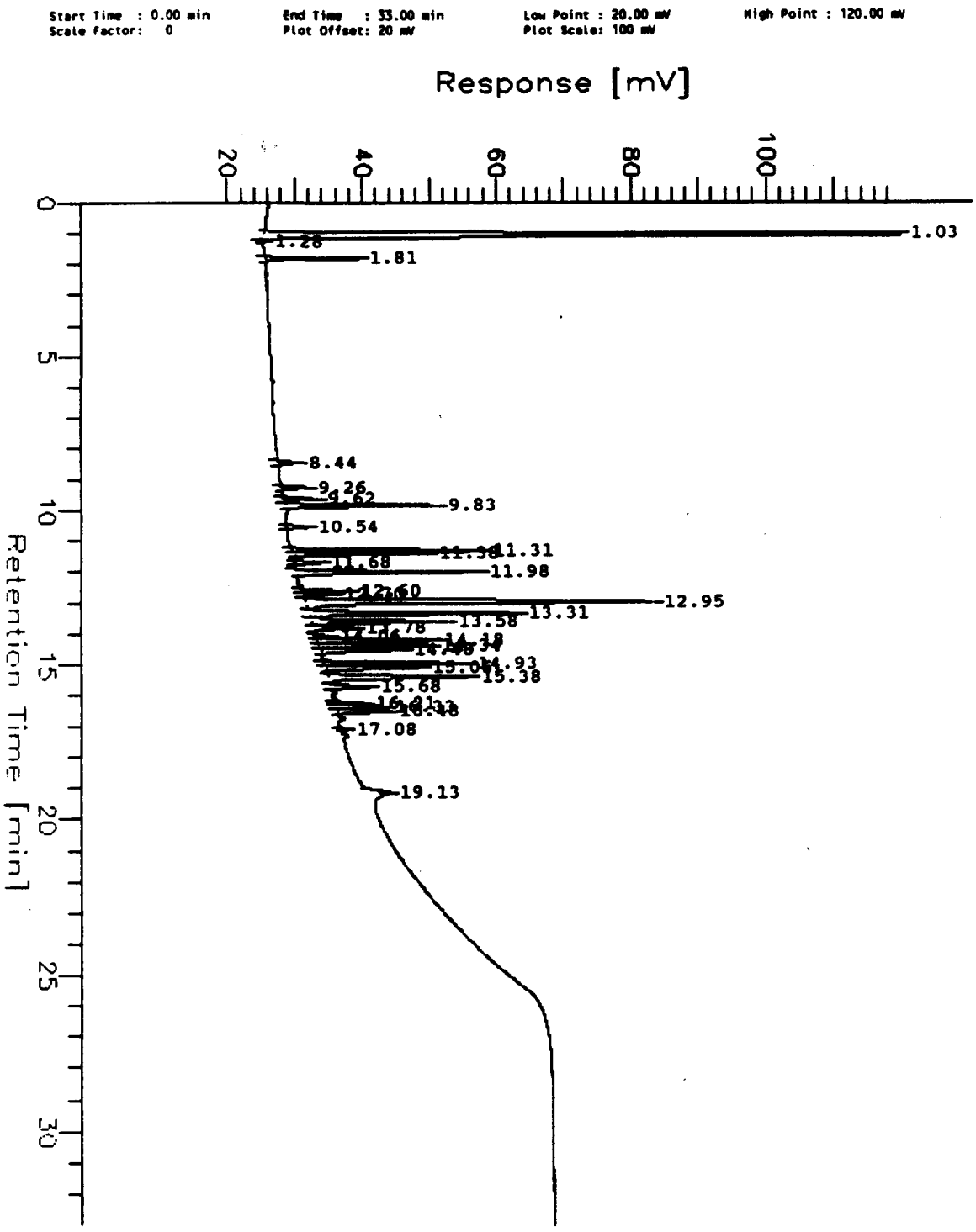
Column: 30 m x 0.25 mm ID, DB-5
Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C; carrier He at 16 psi.

FIGURE 4.
GAS CHROMATOGRAM OF THE TOXAPHENE STANDARD



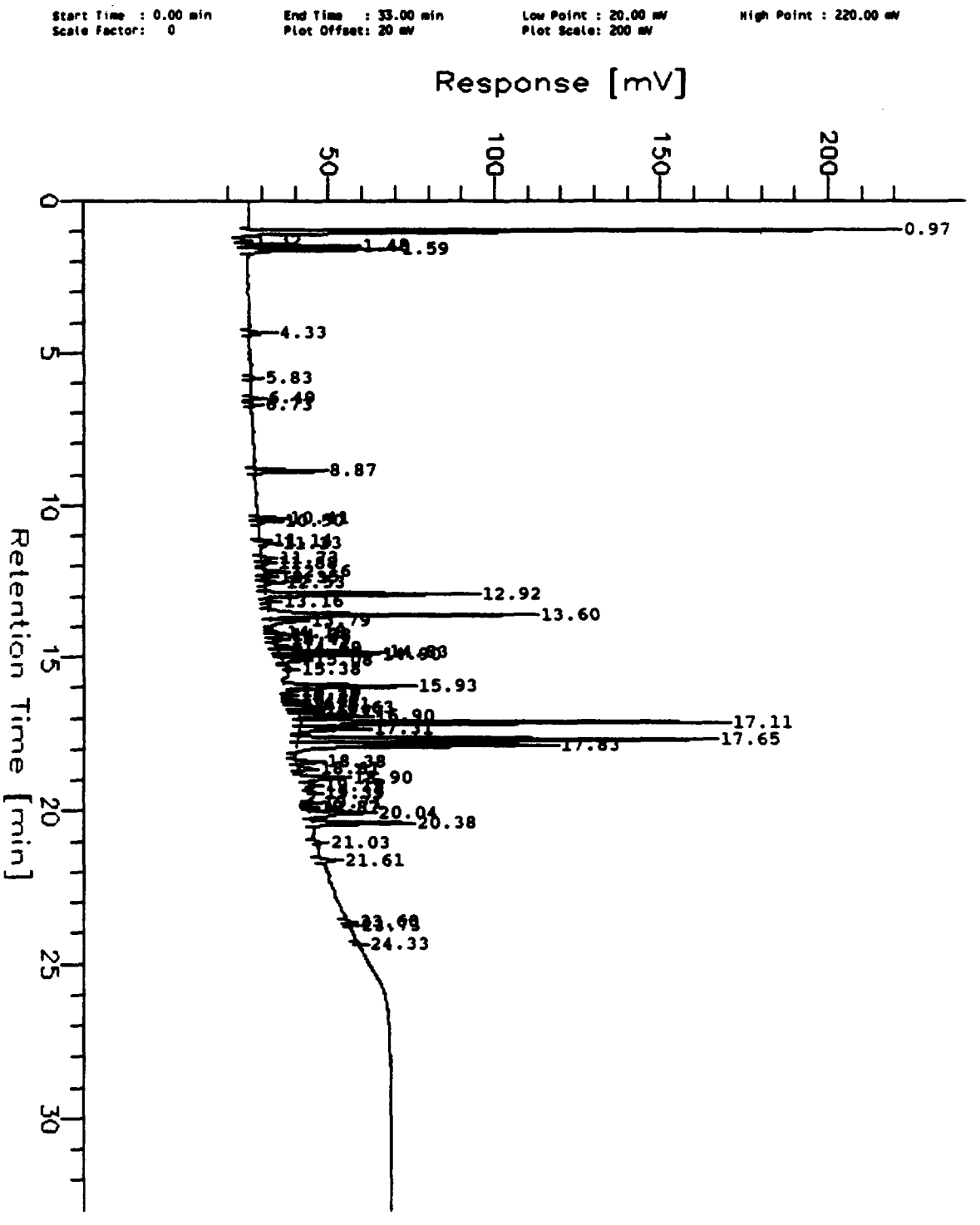
Column: 30 m x 0.25 mm ID, DB-5
Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C; carrier He at 16 psi.

FIGURE 5.
GAS CHROMATOGRAM OF THE AROCLOR-1016 STANDARD



Column: 30 m x 0.25 mm ID DB-5 fused silica capillary.
Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C; carrier He at 16 psi.

FIGURE 6.
GAS CHROMATOGRAM OF THE TECHNICAL CHLORDANE STANDARD



Column: 30 m x 0.25 mm ID DB-5 fused silica capillary.
Temperature program: 100°C (hold 2 minutes) to 160°C at 15°C/min, then at 5°C/min to 270°C; carrier He at 16 psi.

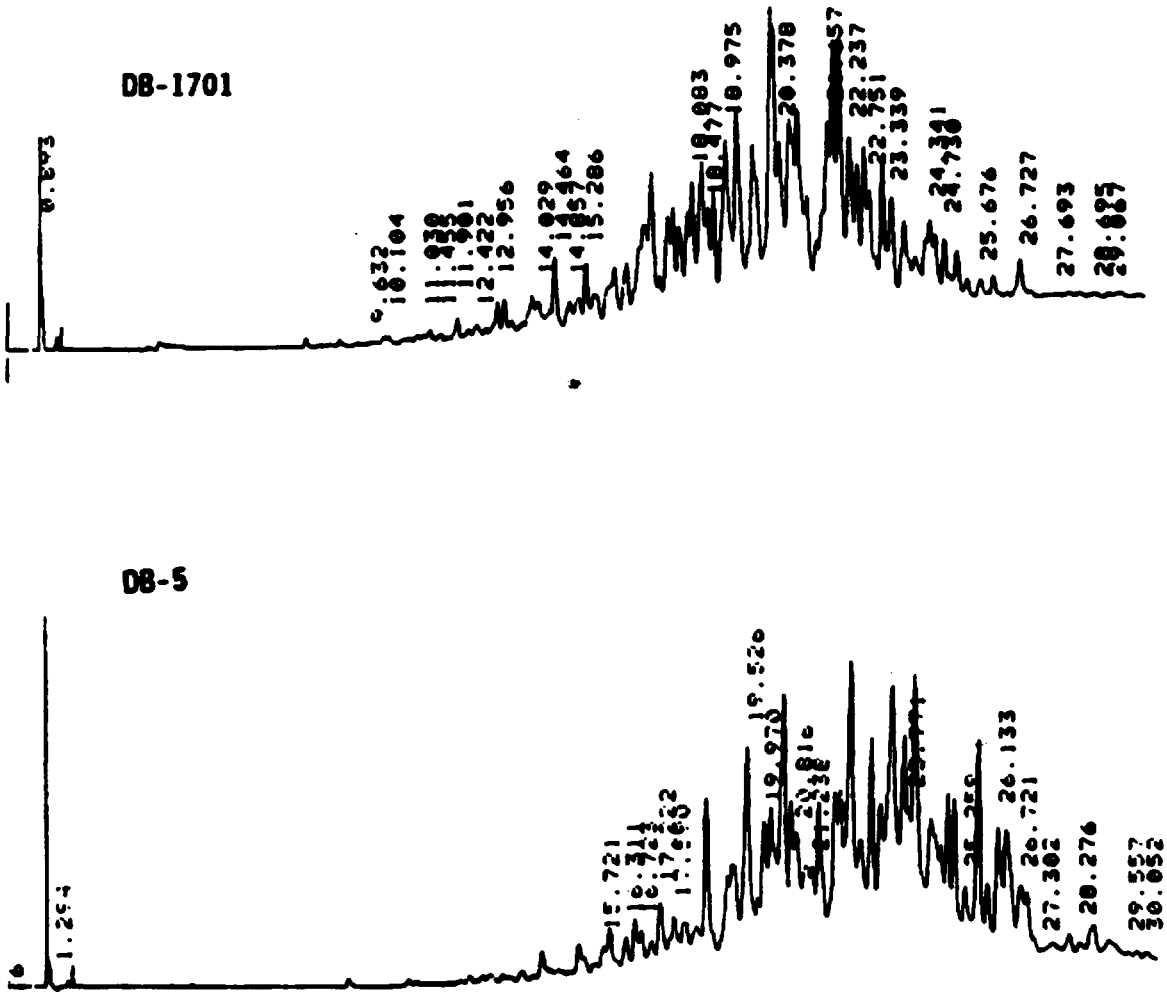


FIGURE 7. GC/ECD chromatogram of Toxaphene analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

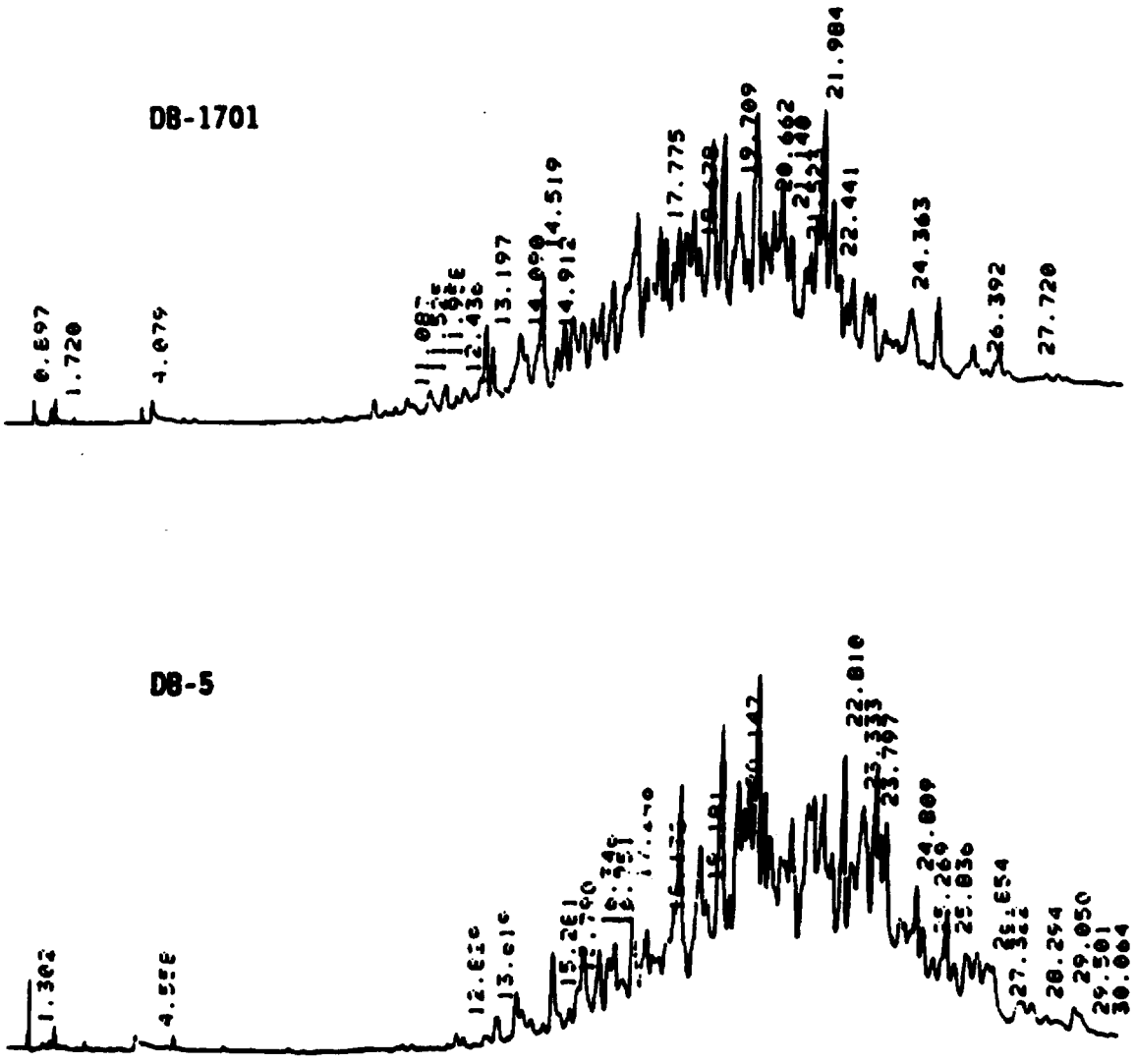


FIGURE 8. GC/ECD chromatogram of Strobane analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

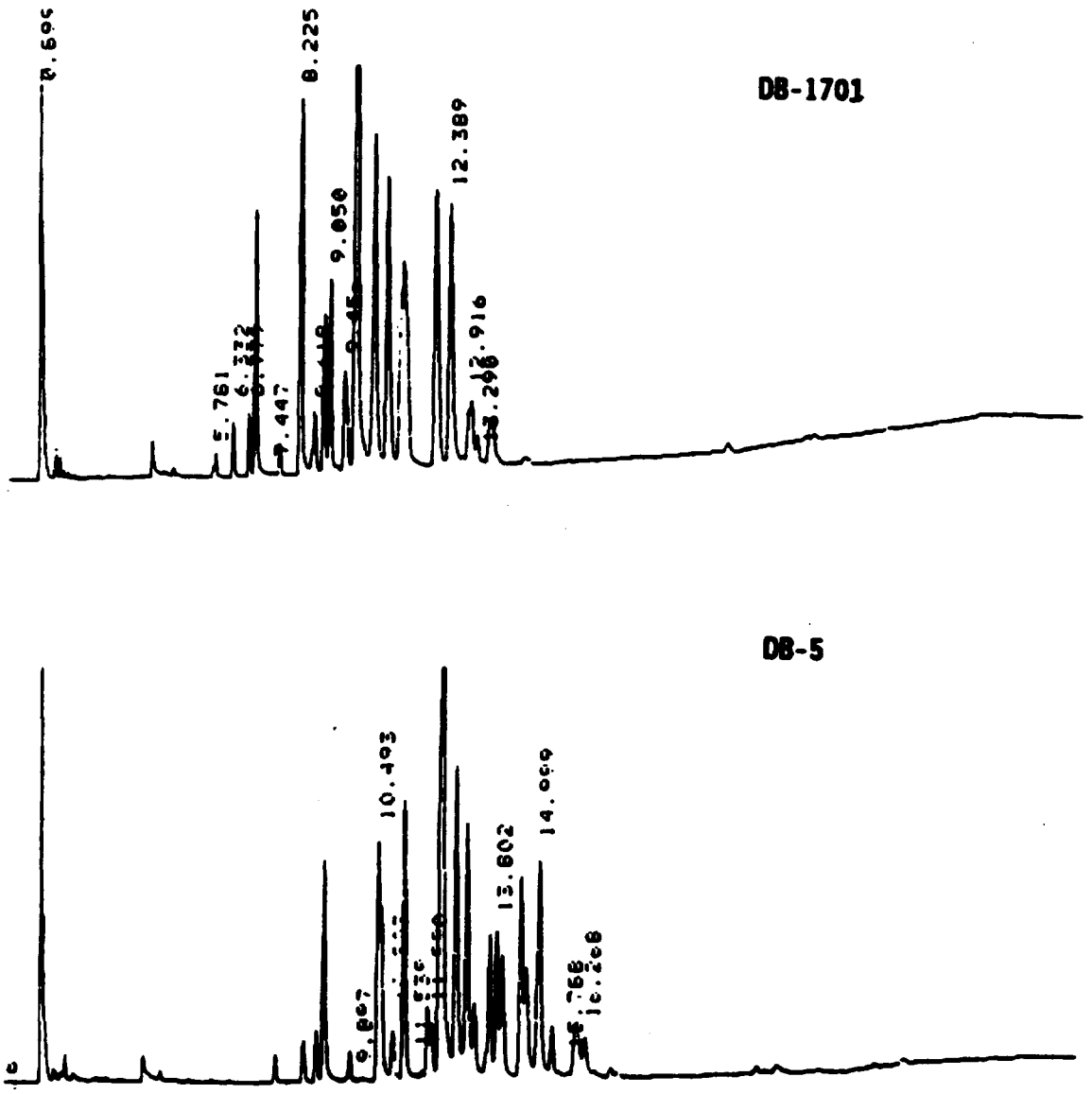


FIGURE 9. GC/ECD chromatogram of Aroclor 1016 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

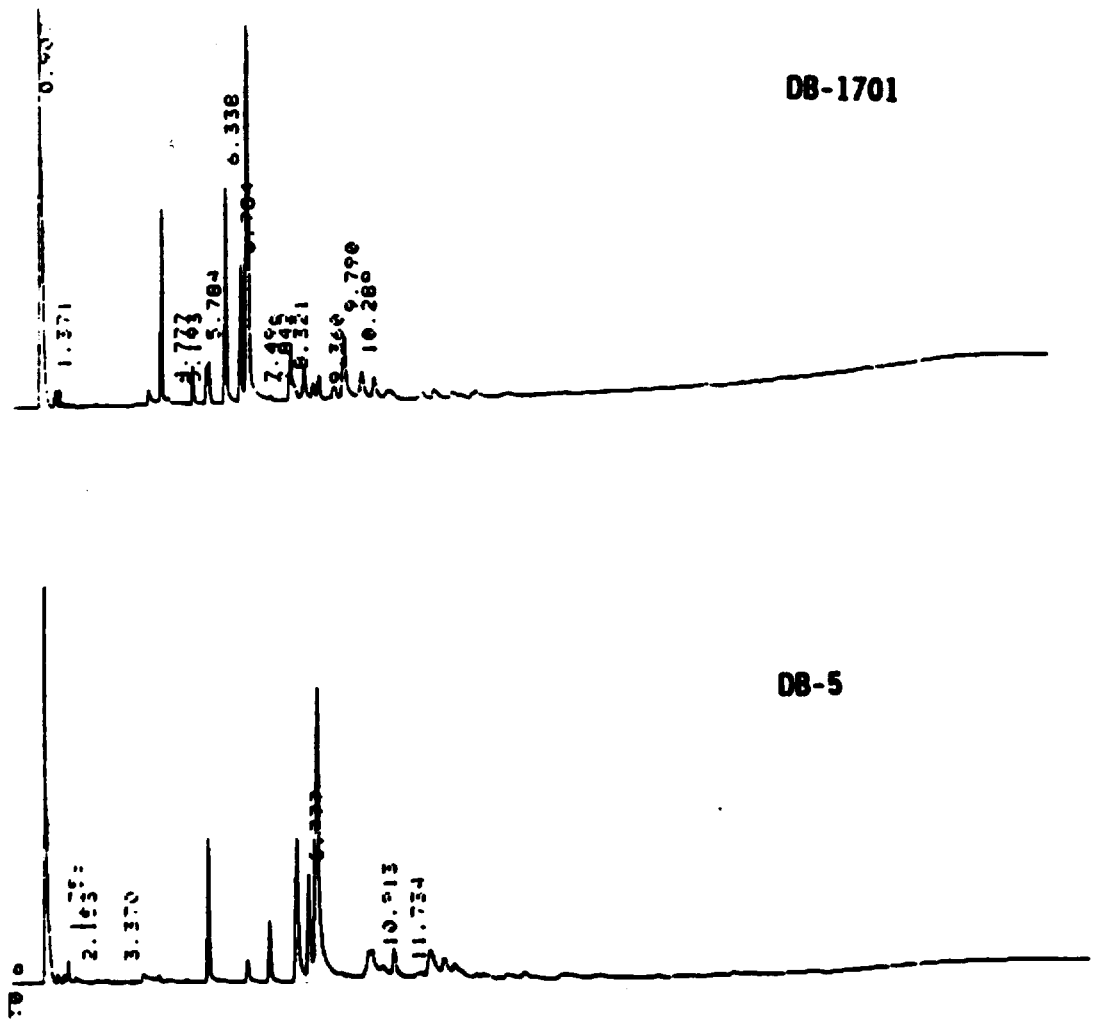


FIGURE 10. GC/ECD chromatogram of Aroclor 1221 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

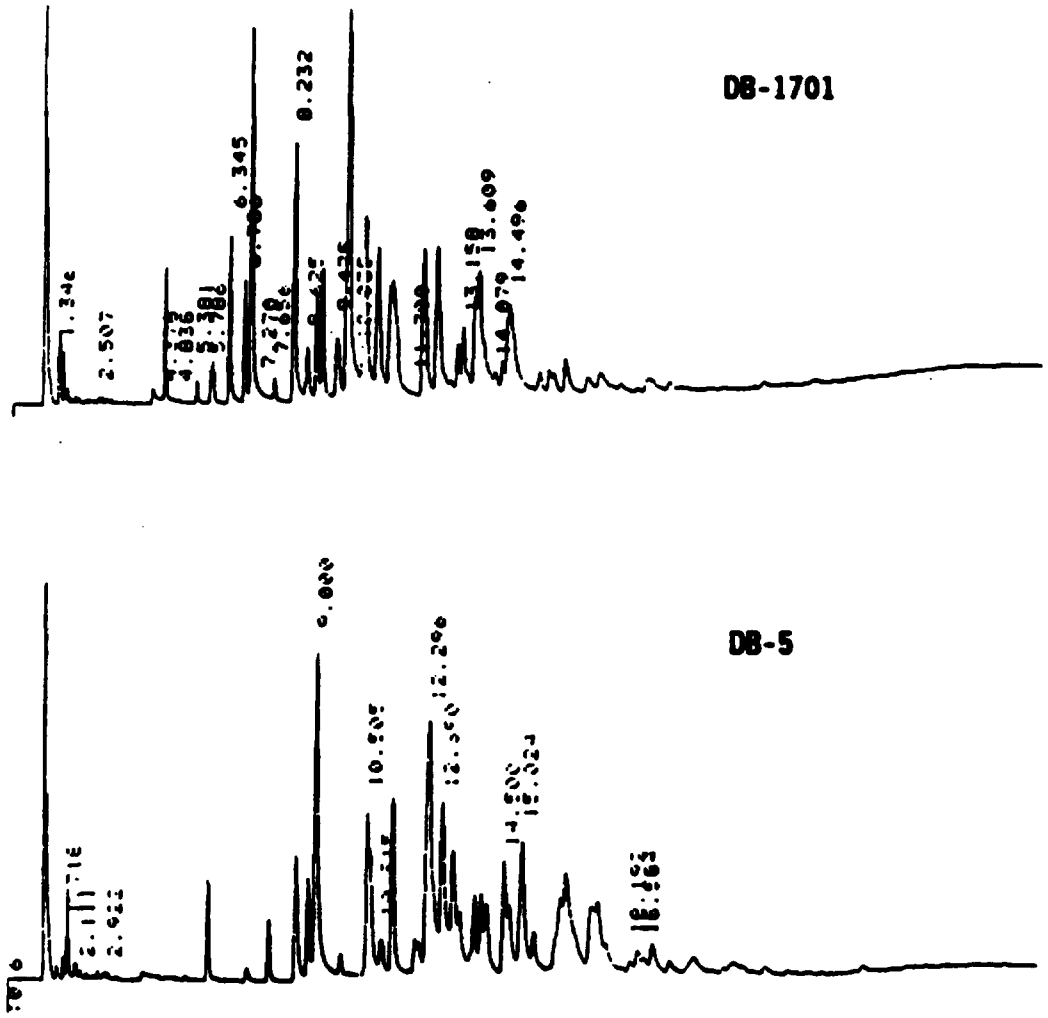


FIGURE 11. GC/ECD chromatogram of Aroclor 1232 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

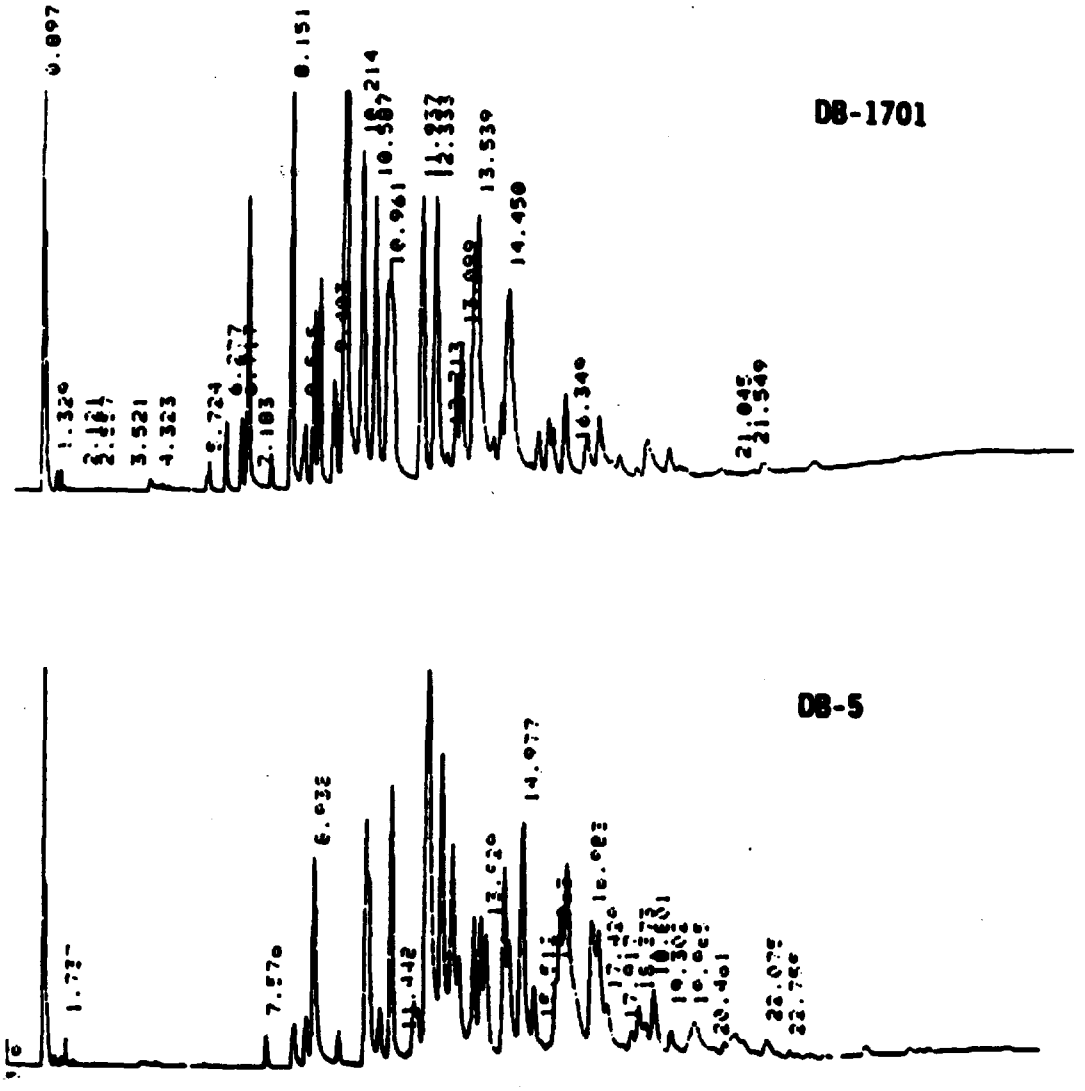


FIGURE 12. GC/ECD chromatogram of Aroclor 1242 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

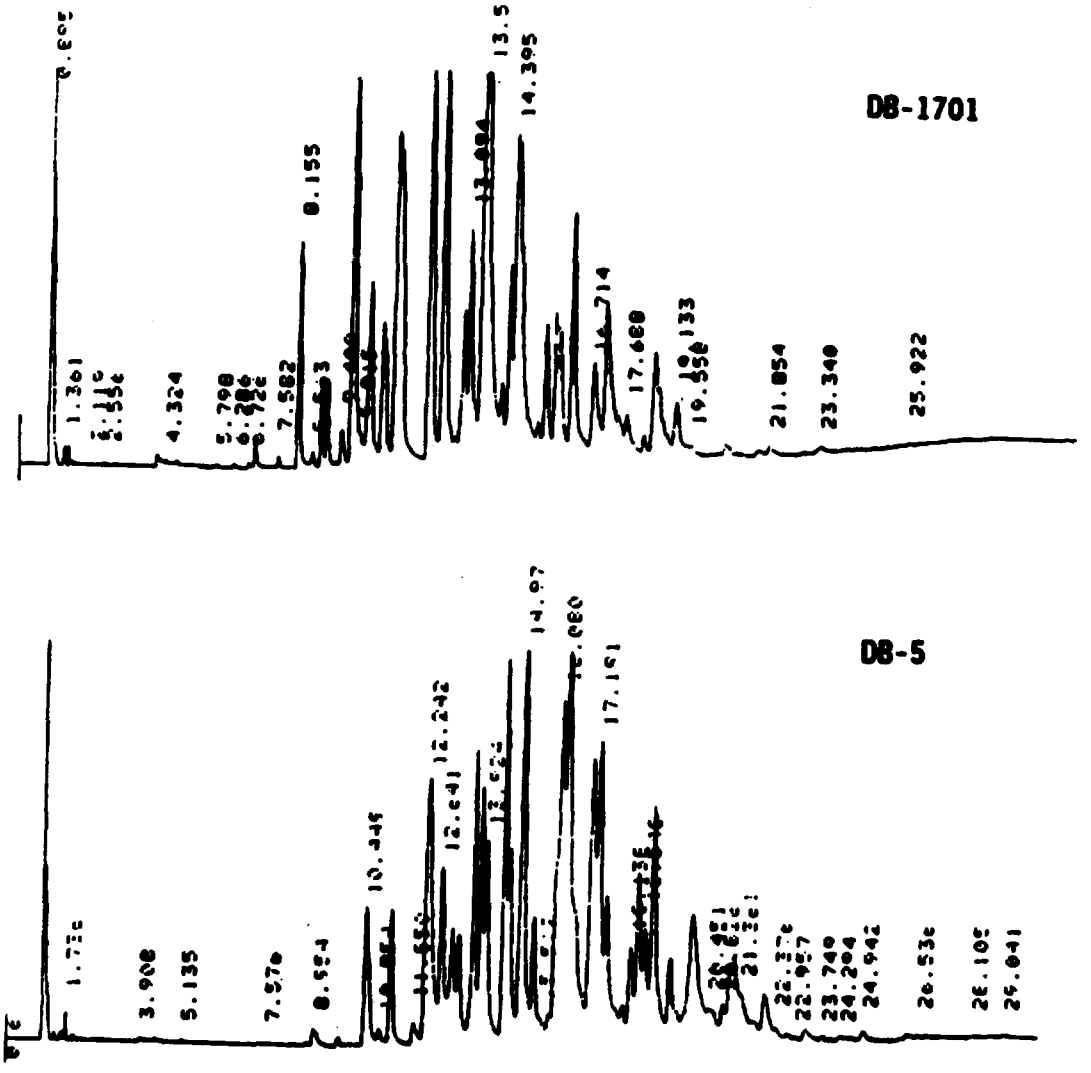


FIGURE 13. GC/ECD chromatogram of Aroclor 1248 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

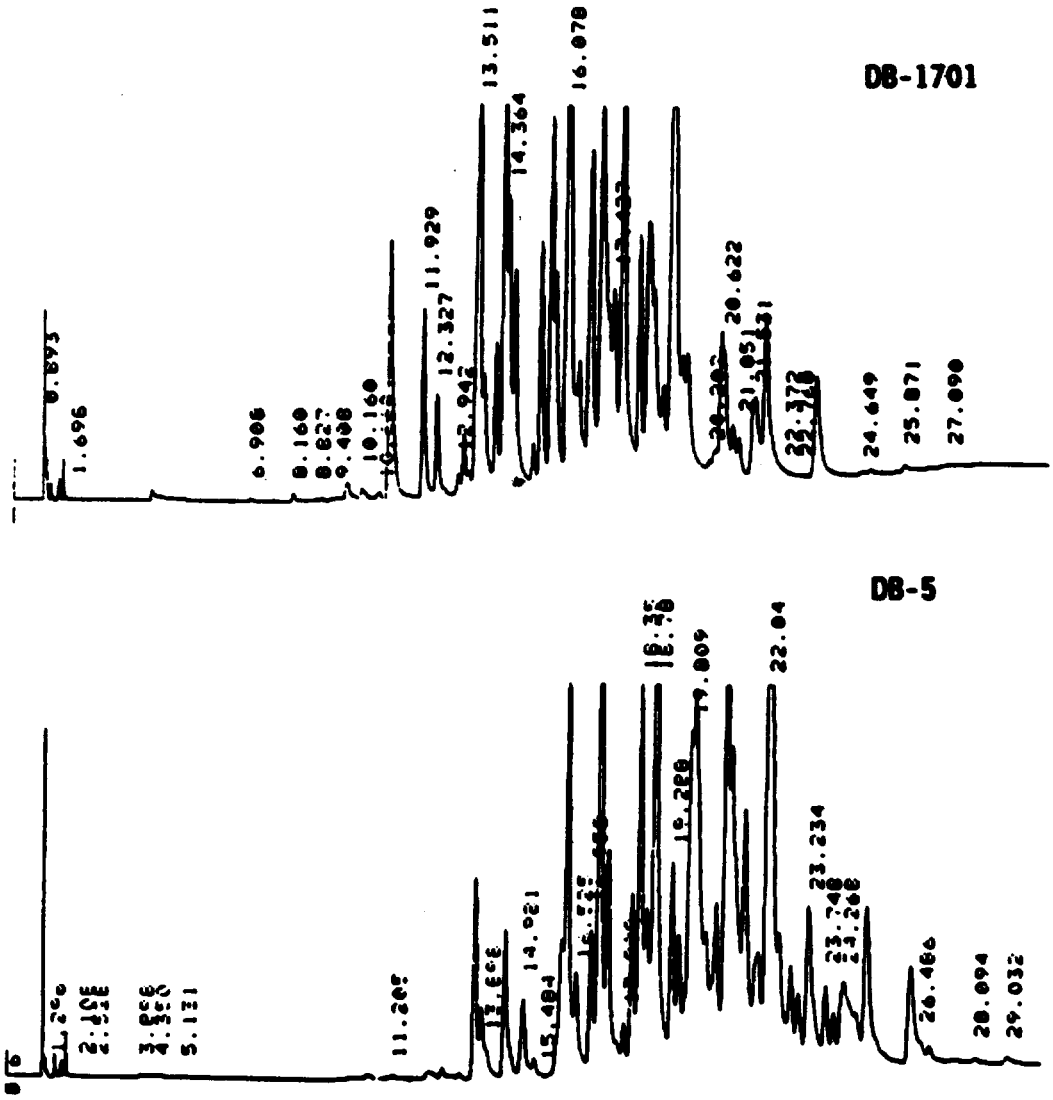


FIGURE 14. GC/ECD chromatogram of Aroclor 1254 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

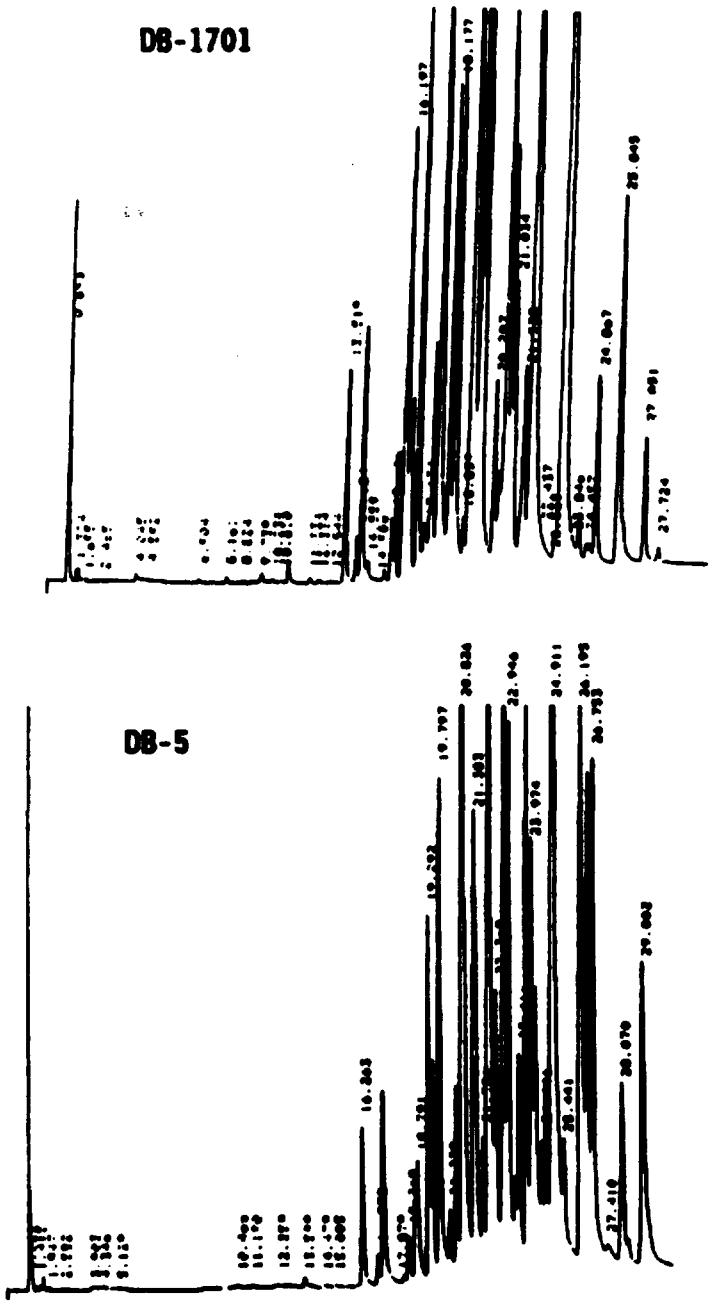


FIGURE 15. GC/ECD chromatogram of Aroclor 1260 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

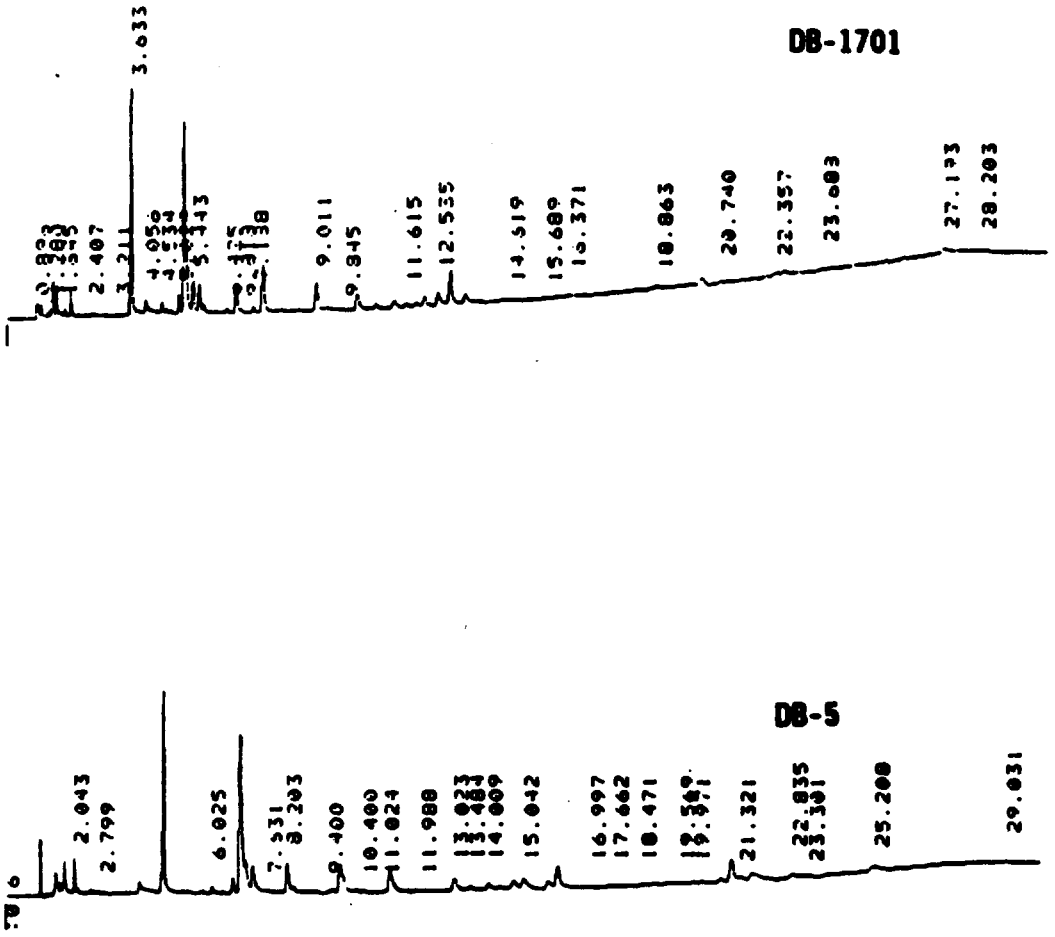


FIGURE 16. GC/ECD chromatogram of Halowax 1000 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

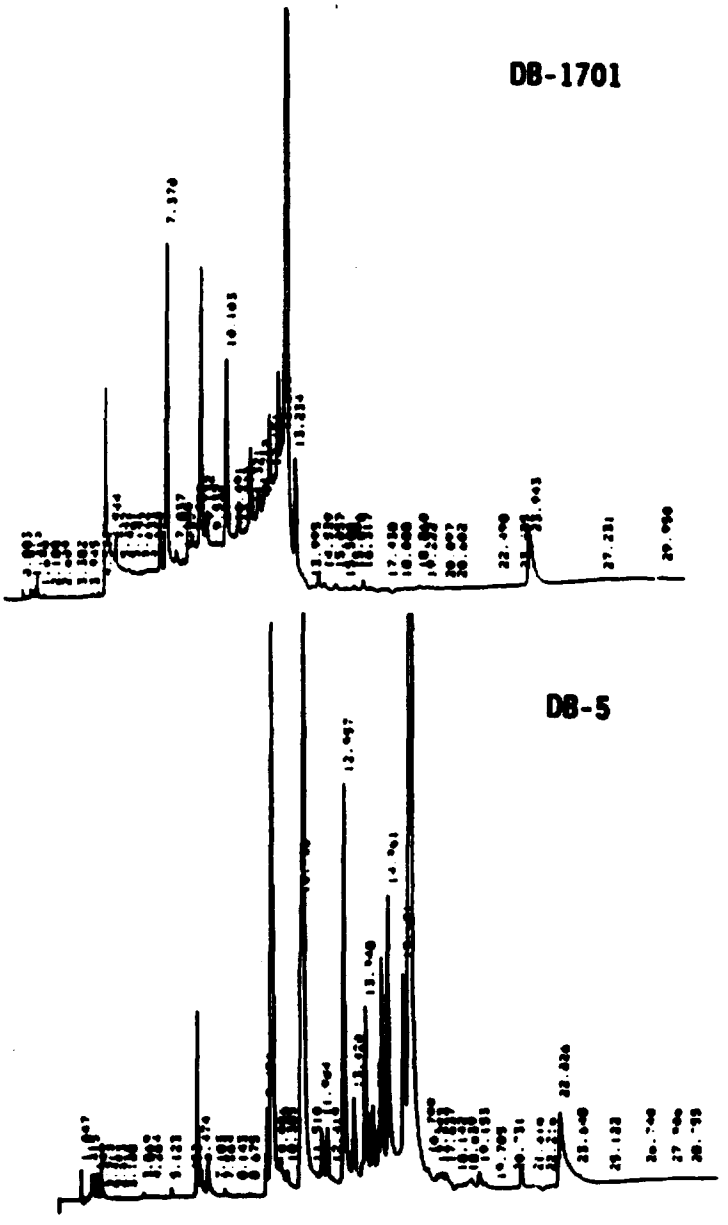


FIGURE 17. GC/ECD chromatogram of Halowax 1001 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

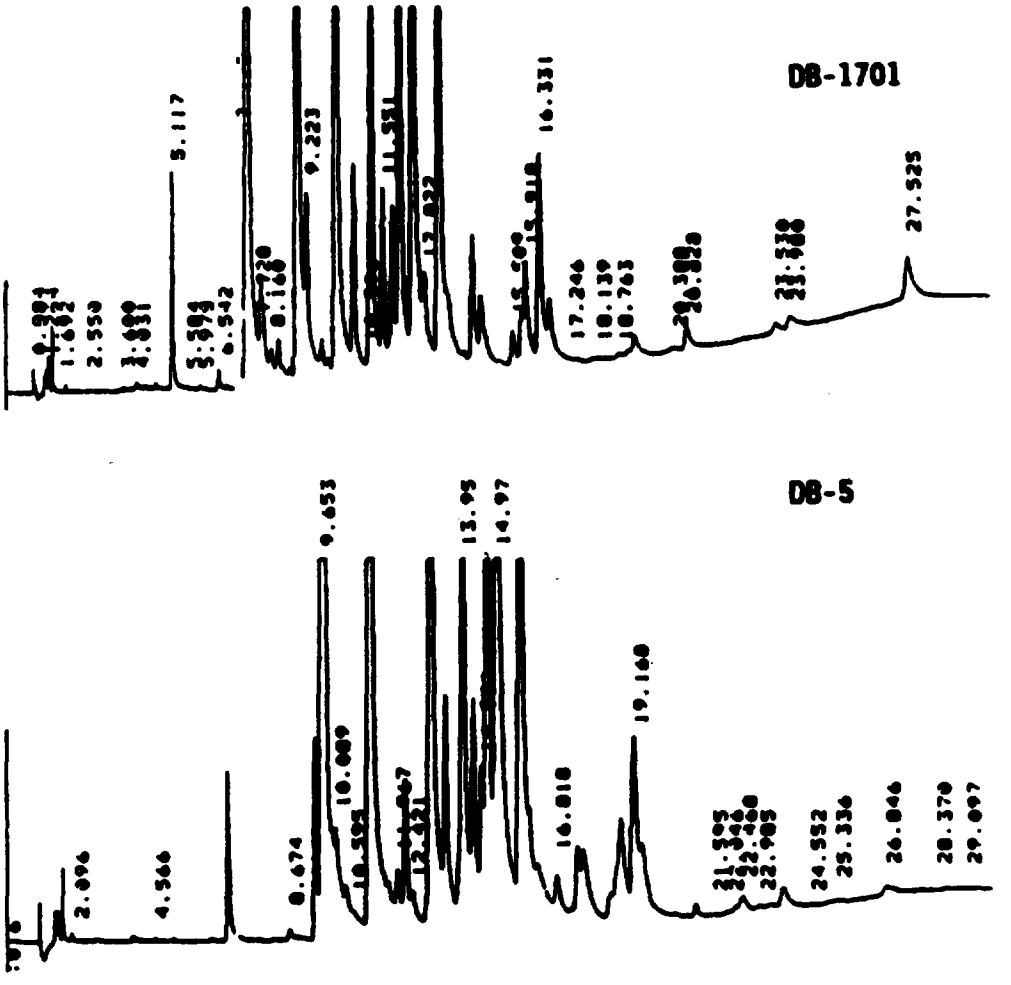


FIGURE 18. GC/ECD chromatogram of Halowax 1099 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

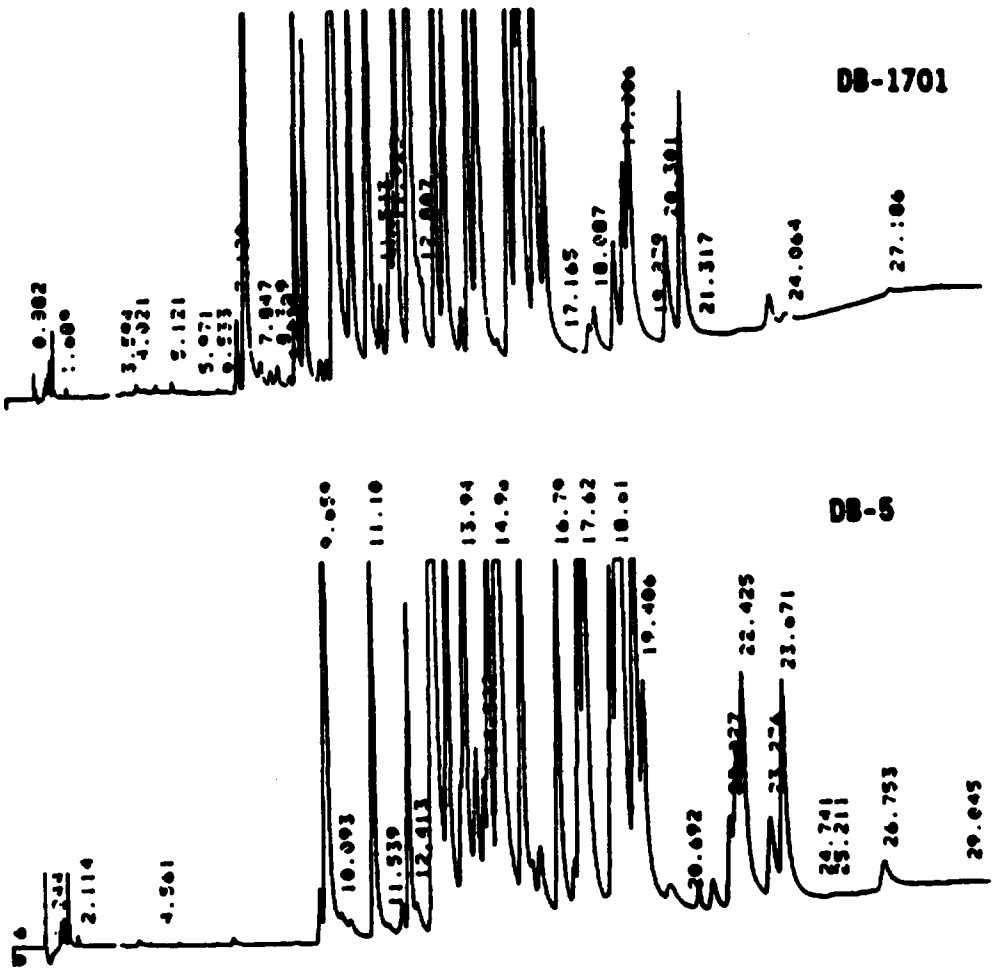


FIGURE 19. GC/ECD chromatogram of Halowax 1013 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

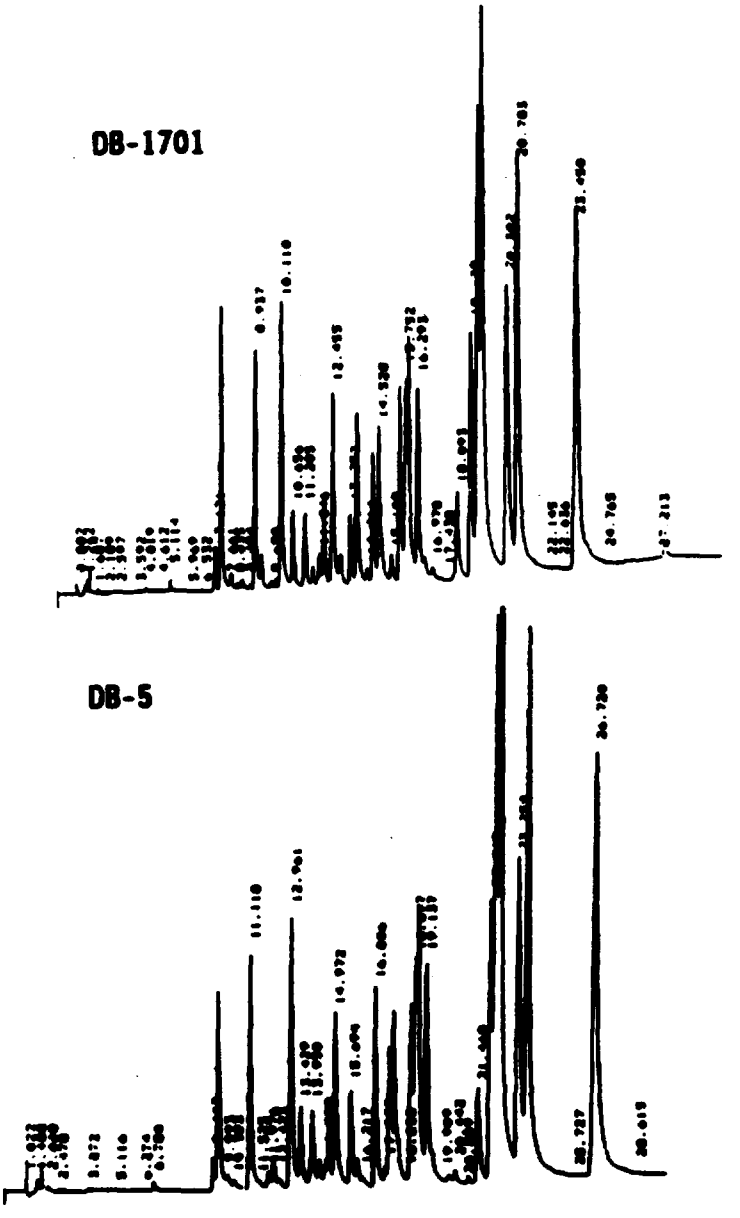


FIGURE 20. GC/ECD chromatogram of Halowax 1014 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

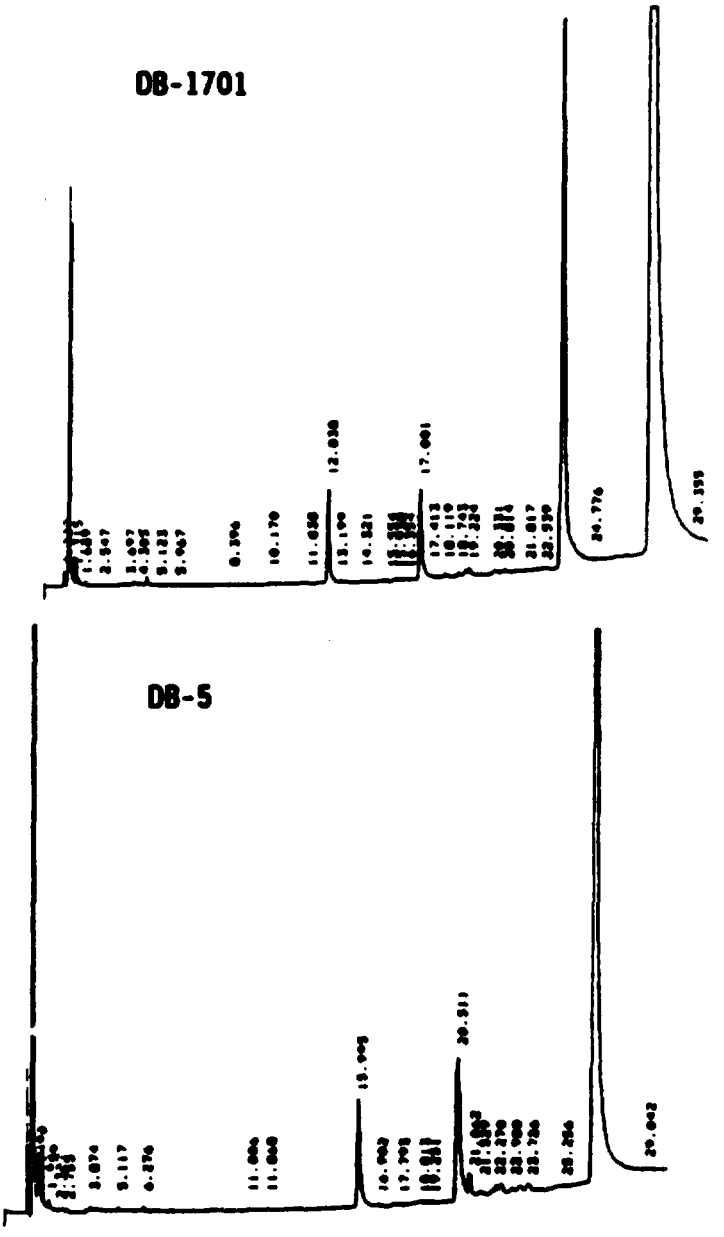


FIGURE 21. GC/ECD chromatogram of Halowax 1051 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

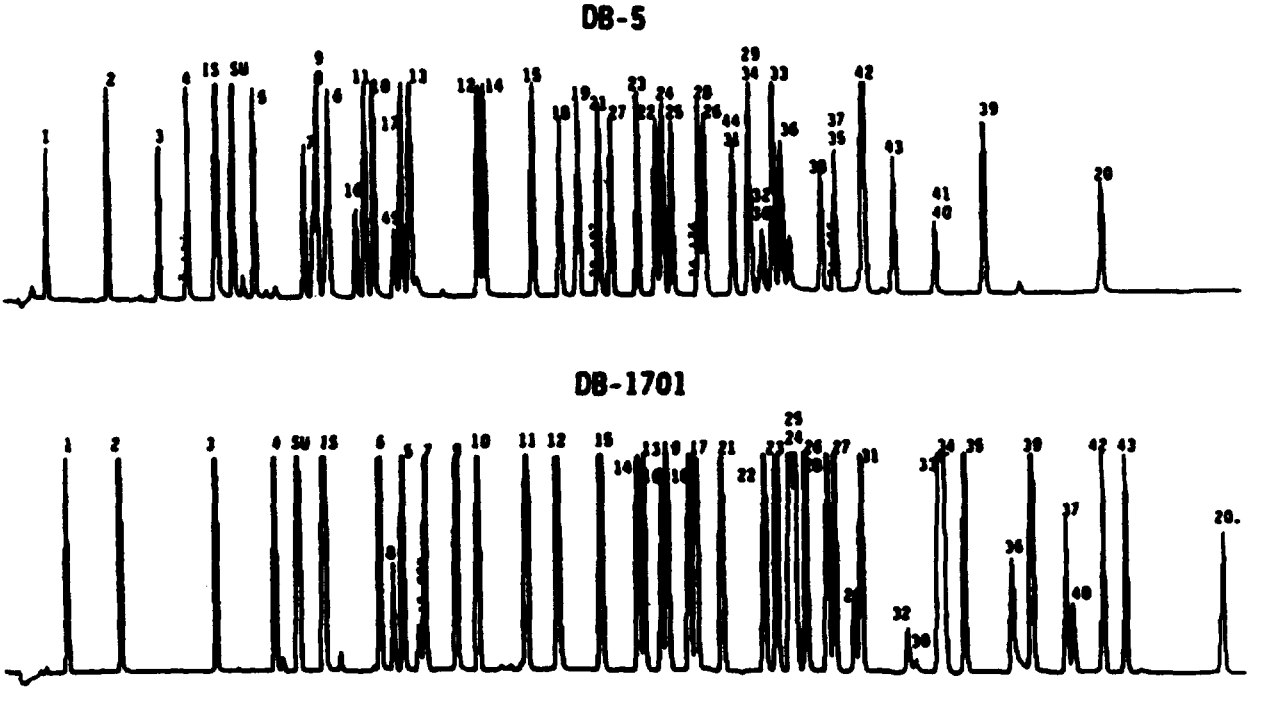


FIGURE 22. GC/ECD chromatogram of the organochlorine pesticides analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (0.83-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to an 8 in injection tee (Supelco Inc.). Temperature program: 140°C (2 min hold) to 270°C (1 min hold) at 2.8°C/min.

ORGANOCHLORINE PESTICIDES AND PCBs AS AROCLORS BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE

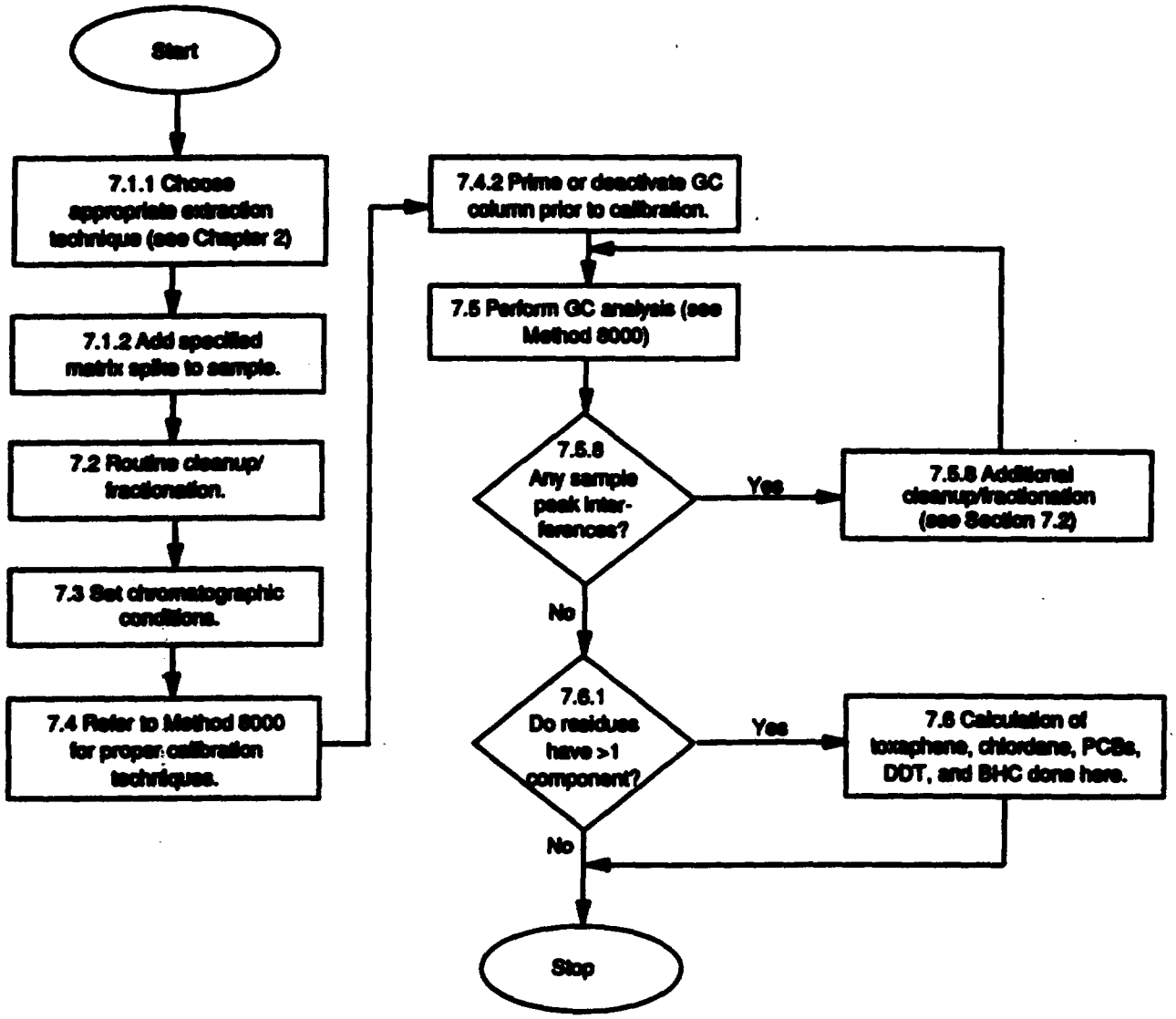


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Figure 9. GC/ECD chromatogram of Aroclor 1016 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 10. GC/ECD chromatogram of Aroclor 1221 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 11. GC/ECD chromatogram of Aroclor 1232 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 12. GC/ECD chromatogram of Aroclor 1242 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 13. GC/ECD chromatogram of Aroclor 1248 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 14. GC/ECD chromatogram of Aroclor 1254 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 15. GC/ECD chromatogram of Aroclor 1260 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 16. GC/ECD chromatogram of Halowax 1000 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 17. GC/ECD chromatogram of Halowax 1001 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 18. GC/ECD chromatogram of Halowax 1099 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 19. GC/ECD chromatogram of Halowax 1013 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 20. GC/ECD chromatogram of Halowax 1014 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 21. GC/ECD chromatogram of Halowax 1051 analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (1.5-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to a J&W Scientific press-fit Y-shaped inlet splitter. Temperature program: 150°C (0.5 min hold) to 190°C (2 min hold) at 12°C/min then to 275°C (10 min hold) at 4°C/min.

Figure 22. GC/ECD chromatogram of the organochlorine pesticides analyzed on a DB-5/DB-1701 fused-silica open-tubular column pair. The GC operating conditions were as follows: 30 m x 0.53 mm ID DB-5 (0.83-μm film thickness) and 30 m x 0.53 mm ID DB-1701 (1.0-μm film thickness) connected to an 8 in injection tee (Supelco Inc.). Temperature program: 140°C (2 min hold) to 270°C (1 min hold) at 2.8°C/min.